140948 ORIGINAL (Red)

HORSEHEAD RESOURCE DEVELOPMENT COMPANY, INC.
PALMERTON, PENNSYLVANIA

QUALITY ASSURANCE PROJECT PLAN FOR ADDITIONAL STUDIES FOR REMEDIAL ACTION AT CINDER BANK OPERABLE UNIT (OPERABLE UNIT 2 - PALMERTON ZINC NPL SITE) SUPPLEMENTAL AIR MONITORING

GAI CONSULTANTS, INC.
570 BEATTY ROAD
MONROEVILLE, PENNSYLVANIA 15146

PROJECT 92-118

JULY 1994

ORIGINAL (Red)

HORSEHEAD RESOURCE DEVELOPMENT COMPANY, INC.
PALMERTON, PENNSYLVANIA

TITLE AND APPROVAL PAGE

FOR

QUALITY ASSURANCE PROJECT PLAN FOR ADDITIONAL STUDIES FOR REMEDIAL ACTION AT CINDER BANK OPERABLE UNIT (OPERABLE UNIT 2 - PALMERTON ZINC NPL SITE) SUPPLEMENTAL AIR MONITORING

JULY 1994

Precha Yodnane — 7/27/94

Precha Yodnane, Ph.D., P.E., Project Manager — Date
GAI Consultants, Inc.

John Oyler — 8-10-94

John A. Oyler, Project Manager — Date
Horsehead Resource Development Company, Inc.

Frederick N. MacMillan, Remedial Project Manager — Date
U.S. EPA Region III

ORIGINAL (Red)

TABLE OF CONTENTS

gai

ORIGINAL (Red)

TABLE OF CONTENTS (Continued)

TABLE OF CONTENTS (Continued)

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

1.0 PROJECT DESCRIPTION

1.1 BACKGROUND

The Palmerton Cinder Bank CERCLA Operable Unit No. 2 consists of approximately 33 million tons of slag-like residues and various related wastes from over 80 years of metal smelting operations. The Cinder Bank also contains a relatively small volume of municipal solid wastes (estimated to be approximately 0.3 percent by weight) from Palmerton and surrounding communities which were co-disposed with the smelting residues for about 55 years. The 2.5-mile long Cinder Bank is situated parallel to and on the foot of Blue Mountain, and is oriented in an east-west direction downwind (south) of the Palmerton Zinc East Plant.

According to the Record of Decision (ROD)[1*], the residues have little vegetal cover to control wind and water erosion, are standing at marginally stable slopes, contain zinc, lead and cadmium, and, in some locations, appear to be smoldering. Regulatory agencies, Horsehead Resource Development Corporation (HRD) and its associate company, Zinc Corporation of America (ZCA), and others are concerned about air pollution from fires burning within the Cinder Bank and the potential for environmental damage due to wind and water erosion of heavy-metal-containing Cinder Bank residue particles, and water pollution from leaching of heavy metals out of the residues by percolate water, run-on of surface water from Blue Mountain, and infiltration of spring water.

*See List of References

In 1985, The United States Environmental Protection Agency (EPA) and the New Jersey Zinc Company negotiated a Consent Decree to conduct the Cinder Bank Remedial Investigation/Feasibility Study (RI/FS)[2]. The 1988 ROD mandated implementation of a conceptual remediation plan for the Cinder Bank. The remedial objectives are to:

1. Minimize direct contact with Cinder Bank residues.
2. Divert Blue Mountain runoff to prevent run-on water from infiltrating and eroding the Cinder Bank.
3. Reduce contaminant levels in runoff from the Cinder Bank.
4. Collect and treat runoff and seeps.
5. Reduce windborne contaminant releases from the Cinder Bank.
6. Control internal fires within the Cinder Bank.

Specific actions called for in the ROD include:

1. Controlling fires to reduce air pollution;
2. Re-grading of the Cinder Bank to more stable slopes to reduce erosion by wind and water;
3. Diverting and managing surface water to reduce run-on from Blue Mountain and to collect and treat Cinder Bank runoff;
4. Capping (with clay and soil) and revegetating the Cinder Bank to reduce the infiltration of water into the residues, reduce wind and water erosion of residues, and isolate the residues from the public.

Since the Consent Decree was issued in 1985, additional studies of the Cinder Bank fires, successful field scale implementation of HRD's Ecoloam™ fly ash/sludge innovative capping

gai

technology on Blue Mountain, and preliminary residue recycling feasibility studies have suggested that the goals of the ROD might be met using more cost-effective measures than those specified in the ROD. In December 1991, EPA issued an Amendment to the Consent Decree[3] that authorizes HRD and ZCA to conduct additional studies of alternative technologies with which to meet ROD objectives.

Additional studies required by the Amendment are:

Item 1) Monitor emissions from Cinder Bank fires to determine whether they pose a threat to public health or the environment;

Item 2) Using hydrologic models, evaluate the effectiveness of the clay/soil cap prescribed in the ROD and alternative cap and cover scenarios, including use of an Ecoloam™ fly ash/sludge cap as both a growing medium and water barrier;

Item 3) Define the vertical and horizontal extent of fires within the Cinder Bank;

Item 4) Recommend fire control measures, if necessary, including locating fire cut-off trench(es); and,

Item 5) Evaluate the feasibility of recycling the Cinder Bank residues to recover metal and/or energy values in the residues while also meeting environmental objectives of the ROD.

HRD has retained GAI Consultants, Inc., (GAI) to conduct study Items 1 to 4 according to the Amendment to the Consent Decree.

An air monitoring program to address Item 1 (above) was conducted between November 17, 1992 and December 9, 1992. The results of this study, as presented in the GAI

Air Monitoring Report dated April 1993[4], were inconclusive for benzene, sulfur dioxide (SO_2) and hydrogen sulfide (H_2S).

This Quality Assurance Project Plan (QAPP) has been prepared as an addendum to the Blue Mountain Operable Unit QAPP[5] and GAI Cinder Bank QAPP[6] to address the specific and unique project and quality requirements of the supplemental air monitoring required for benzene, SO_2 and H_2S on the Cinder Bank Operable Unit. This QAPP details the quality control procedures that will be implemented to maintain the quality assurance of the field activities associated with the completion of these additional studies on the Cinder Bank Operable Unit in Palmerton, Pennsylvania. Where applicable, the quality assurance versus quality control roles and procedures for this program are identified as such throughout this document. The definition for quality assurance and quality control, as stated by the U.S. Environmental Protection Agency[7] are as follows:

- Quality Assurance: The total program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.
- Quality Control: The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

1.2 PROJECT OBJECTIVES

The intent of the 1988 ROD and subsequent request for additional studies was: (1) to reduce contamination of surface- and ground-water associated with uncontrolled run-on, runoff, erosion, and infiltration; (2) reduce the potential for air pollution associated with Cinder Bank

wind erosion and/or fires; and, (3) reduce the potential for direct contact with Cinder Bank residues. The additional studies presented in this work plan, as required by the 1991 Amendment to the Consent Decree, incorporate the experience of recent years and identify innovative and more cost-effective remediation approaches for the Cinder Bank.

Objectives of these studies are:

1) Air monitoring to determine whether smoldering fires within the Cinder Bank are a hazard to public health or the environment.

2) Modeling the performance of various Cinder Bank caps, including the clay/soil cap stipulated in the ROD, an innovative alternative using Ecoloam™ sludge/fly ash mixture proposed by HRD, and other capping systems to determine the adequacy of capping alternatives.

3) Delineation of the vertical and horizontal extent of the fires within the Cinder Bank and evaluation of potential fire control measures.

4) Identifying locations of fire cut-off trench(es) if appropriate.

An air monitoring program to address Item 1 was conducted between November 17, 1992 and December 9, 1992. Based on the results of this study, as presented in the GAI Air Monitoring Report dated April 1993, the following conclusions were made:

- Cinder Bank vent emissions are not a source of airborne Total Suspended Particulate (TSP), heavy metals, benzene, or Polyaromatic Hydrocarbon (PAH) contamination detected at this site.
- Cinder Bank vent emissions present no significant hazard to public health or the environment.

- TSP and heavy metal contamination detected during this study are the result of windblown fugitive particulate from unvegetated areas of the Cinder Bank and vehicular traffic on the Cinder Bank.
- Since airborne contaminants detected at this site are resultant from sources other than Cinder Bank vent emissions additional air monitoring at perimeter and off-site locations is not necessary.

Several problems were encountered with the sampling techniques used in this study for benzene, sulfur dioxide (SO_2), and hydrogen sulfide (H_2S). Extremely low levels of benzene were detected in all areas sampled. This indicated a vestigial contamination inherent in the sampling methodology not related to Cinder Bank vent emissions. Although SO_2 and H_2S were not detected in any area of the Cinder Bank, these results were considered to be inconclusive since the detection limit for the sampling instrumentation was not low enough to satisfy the study objectives for minimum detectable concentration.

Satisfying the study objectives will require fulfilling the data needs and quality objectives detailed in Table 1. A listing of air contaminants to be evaluated during the supplemental air monitoring phase of this project is presented in Table 2.

Additional information concerning this phase of the project, including a more detailed project description, work plan, location site maps, and sampling locations is presented in the GAI "Work Plan for Additional Studies - Supplemental Air Monitoring."[8]

The project schedule is presented in Figure 1.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The organization of the project responsibilities and lines of authority of personnel involved in the additional studies on the Cinder Bank Operable Unit are shown in Figure 2 and described in the following subsections. The resumes of pertinent project personnel are provided in Appendix A.

2.1 PROJECT DIRECTOR (QUALITY ASSURANCE ROLE)

The Director of the Remediation Services Division of HRD will be responsible for the overall project, including objectives, scope, budget, schedule, and quality of submittals. The Director will promote continuity, report to management, and provide financial assurance regarding quality assurance/quality control (QA/QC).

2.2 PROJECT MANAGER

The HRD Project Manager will be responsible for planning, coordinating, integrating, monitoring, and appraising (i.e., managing) all project activities. These will include the activities of any contractors to HRD.

2.3 QUALITY ASSURANCE MANAGER (QUALITY ASSURANCE AND QUALITY CONTROL ROLE)

The Quality Assurance Manager (QAM) will be responsible for implementation of all aspects of the quality assurance (QA) program described in the QAPP. In addition, the QAM will be responsible for reviewing and approving all modifications to the QA program and

arranging periodic audits to ascertain if the program objectives are being achieved. The duties of the QAM include QA review and approval of sampling procedures and field documentation (such as daily logs and inspections). The QAM will have the authority to impose proper procedures or to halt an operation.

2.4 GAI CONSULTANTS (QUALITY ASSURANCE AND QUALITY CONTROL ROLE)

The GAI Project Manager is responsible for all QA/QC activities related to the work of GAI and their subcontractors.

2.5 LABORATORY MANAGERS (QUALITY ASSURANCE AND QUALITY CONTROL ROLE)

The Laboratory Managers at Lancaster Laboratories, Inc., Lancaster, Pennsylvania will be responsible for implementation, evaluation, and documentation of the QA program at their facility. The responsibilities of the Laboratory Manager include:

- Administering the laboratory QA/QC program.
- Establishing QC procedures for each test parameter.
- Reviewing the sampling and analytical methodology employed by field and laboratory personnel and modifying these protocols, as necessary.
- Coordinating performance auditing.
- Reviewing analytical results, including raw data, calculations, etc.
- Inspecting laboratory logbooks and the data retrieval systems.
- Monitoring the proper documentation and maintenance of records.
- Overseeing the QA/QC implementation on a daily basis.

- Identifying QA/QC problems and recommending appropriate corrective action.

2.6 FIELD TECHNICIANS AND CHEMISTS (QUALITY CONTROL ROLES)

The field technicians and chemists who are responsible for air monitoring have the following QA/QC responsibilities:

- Being familiar with and practicing the policies and procedures set forth on the QA/QC plan.
- Following sampling protocols in the collection of field samples, recording observations and measurements, and sample handling.
- Being proficient in site safety practices and emergency response procedures.
- Conduct routine maintenance and calibration of field sampling equipment.

2.7 LABORATORY CHEMISTS AND TECHNICIANS (QUALITY CONTROL ROLES)

The laboratory chemist and technicians at the off-site laboratories have the following QA/QC responsibilities:

- Being familiar with and following procedures and policies contained in the QA/QC plan.
- Following analytical procedures in the processing, preparation, handling and analysis of each sample.
- Conduct routine maintenance, standardization, and calibration of analytical equipment.
- Reviewing analytical results with the Laboratory Manager.

- Reporting all irregular results or practices to the Laboratory Manager.

2.8 SITE HEALTH AND SAFETY COORDINATOR

The Site Health and Safety Coordinator will be responsible for the following:

- Keeping an up-to-date Health and Safety Plan (HASP) on site.
- Training of all personnel involved in Health and Safety procedures.
- Overseeing that contractors adhere to the HASP.
- Maintaining control in emergencies.
- Setting up and maintaining site files and the site safety log of activities.
- Maintaining safety equipment and calibration document files.
- Maintaining equipment inspection logs.
- Inspecting first aid kits, fire extinguisher, and eye wash/safety showers regularly for proper stock, maintenance and accessibility, and readiness for use.
- Performing site audits/inspections.
- Upgrade level of protection, as necessary.

The Site Health and Safety Coordinator will be the designated HRD contact for employees at the site, any regulatory agencies, and the public concerning any health and safety-related issues.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT

This section provides the QA objectives established to ensure that the measurement data reported in support of this project will be of appropriate and known quality. QA objectives are measured in terms of precision, accuracy, representativeness, completeness, and comparability (PARCC). The PARCC parameters are indicators of data quality. The nature of this project limits the ability to assign strict PARCC goals. The overall PARCC goals for this work are based on the published precision and accuracy information for the sampling and analytical methods employed, where available, and laboratory derived data.

Since air monitoring samples cannot be replicated, duplicates and spikes will not be performed. Precision and accuracy for quality control, will be measured by results of control samples analyzed concurrently with the field samples.

A summary of quality objectives for air contaminant monitoring are detailed in Table 2.

4.0 FIELD SAMPLING PROCEDURES

4.1 INTRODUCTION

This section contains a summary description of sampling procedures to be used during the field activities of this project. Specific details concerning sampling site selection and description are provided in the GAI Work Plan[8].

4.2 AIR MONITORING STUDY

Air monitoring for the target contaminants will be conducted using the sampling methods listed in Table 2 and discussed in the following subsections.

4.2.1 Benzene

Air sampling for benzene will be conducted using EPA Standard Method T0-14, "Method for the Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using SUMMA^R Passivated Canister Sampling and Gas Chromatographic Analysis," (Appendix B-1). This method involves the collection of contaminant in a SUMMA^R gas sampling canister. Clean, evacuated SUMMA^R canisters will be provided by Lancaster Laboratories, Inc.

4.2.2 Sulfur Dioxide

Air sampling for sulfur dioxide (SO_2) will be conducted using a TEI Model 43A Pulsed Fluorescence SO_2 Analyzer. This is a direct-reading continuous monitor that is approved by EPA for ambient air monitoring (EPA approval - EQSA-0486-060). Standard operating procedures are presented in Appendix C-1.

gai

4.2.3 Hydrogen Sulfide

Air sampling for hydrogen sulfide (H_2S) will be conducted using a TEI Model 45 Pulsed Fluorescence H_2S analyzer. This is a direct reading continuous monitor composed of a TEI Model 43A Pulsed Fluorescence SO_2 Analyzer preceded by a TEI Model 340 H_2S Converter. Standard operating procedures are presented in Appendix C-2.

4.3 PERSONNEL AIR MONITORING

Personnel air monitoring will be conducted during site activities to assure employee safety as detailed in the GAI Site Health and Safety Plan for Cinder Bank studies[9]. This monitoring will be conducted using direct-reading continuous monitors. The specific parameters to be monitored, monitoring equipment, and site action levels are detailed in Table 3. Standard operating procedures for the equipment used for personnel air monitoring are included in Appendix C.

5.0 SAMPLE IDENTIFICATION, DOCUMENTATION AND CUSTODY

5.1 SAMPLE IDENTIFICATION AND DOCUMENTATION

Each sample container will be labeled with the following information:

- Unique sample identification number.
- Date and time of collection.
- Analysis requested.

After sample collection and before proceeding to the next sampling point, the samplers will complete the following procedures:

- Enter the sample into the Chain-of-Custody Record.
- Place the sample container into a shipping container.
- Apply signed custody seals on opposite sides of the shipping container lid in such a manner that the container can not be opened without cutting through the tape.

Custody seals or any other tape must not be used on the lids of the sample containers themselves. The glue used in the tape adhesive may contaminate the sample.

A bound field notebook will be maintained by the lead Field Technician at the site to record daily activities, including sample collection and tracking information. Entries will be made in waterproof ink. A separate entry will be made for each sample collected. Entries will include at least the following information:

- Sample identification number.
- Sample location.
- Date and time of collection.

In-situ field measurements from direct-reading instrumentation and equipment, such as air monitors and meteorological measurements, will also be recorded in the bound field notebook in the same manner.

5.2 SAMPLE IDENTIFICATION

Unique sample identification numbers will be assigned in such a manner that the project, specific task, and individual sample may be identified. The sample identification scheme is presented in Table 4.

5.3 SAMPLING MEDIA AND CONTAINERS

All sampling media required for air monitoring, such as SUMMA® canisters and shipping containers for such media will be provided by Lancaster Laboratories, Inc. All such sampling media will be prepared, shipped, handled, and preserved, if necessary, in accordance with the referenced EPA sampling and analytical method.

5.4 CHAIN OF CUSTODY

5.4.1 Introduction

The chain of custody provides a mechanism for tracing the possession, handling and routing of a sample from the time it is secured, through analysis, to admission to court, if necessary. The chain-of-custody process is initiated by the sampler after a sample is collected, identified and preserved and includes recordkeeping and sample labeling, packaging and shipment procedures. It also includes the laboratory logbook in which sample receipt,

preparation, instrument methods, experimental conditions, and QC analysis results are recorded by the laboratory sample custodian and analysts. The chain of custody is the responsibility of the field sampler until the sample is transferred or otherwise dispatched properly. The Project Manager is responsible for determining whether proper custody procedures were followed during field activities.

5.4.2 Sample Custody

A sample is in custody if:

- it is in the field investigator's or the transferee's actual possession; or
- it is in the field investigator's or the transferee's view, after being in his/her physical possession; or,
- it was in the field investigator's or the transferee's physical possession and then he/she secured it to prevent tampering; or,
- it is placed in a secure sample storage area.

5.4.3 Transfer of Custody

Figure 3 is a sample Chain-of-Custody Record. This form is used when transferring the possession of samples. When the possession of samples is transferred, the individuals relinquishing and receiving must sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory.

Samples are packaged for shipment and dispatched to the appropriate laboratory for analyses with a separate custody record accompanying each shipment (for example, one for each field laboratory, and one for samples driven to the main laboratory). Shipping containers are padlocked or sealed for shipment to the laboratory. Custody seals are to be placed on opposite sides of the container lid prior to final packaging for shipment in such a way that the container can not be opened without cutting through the tape. The method of shipment, courier name(s), and other pertinent information are entered in the "Remarks" box on the form.

Whenever samples are split or duplicated for another party or government agency, this action is noted in the "Remarks" section of the form. The note indicates with whom the samples were split and is signed by both the sampler and recipient. If either party refuse a split or duplicate sample, this is noted and signed by both parties. The person relinquishing the samples to the facility or agency should request the signature of a representative of the appropriate party, acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Remarks" space. When appropriate, as in the case when the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

All shipments must be accompanied by the Chain-of-Custody Record identifying the contents. The original record accompanies the shipment, and a copy is retained by the Project Manager for the project file.

If sent by mail, the package should be registered with return receipt requested. It sent by common carrier, a Bill of Lading is used. The name of the carrier (e.g., Air Express) and

the air bill number are entered in the "Remarks" column. Freight bills, post office receipts, and Bills of Lading should be retained for the project file.

6.0 CALIBRATION PROCEDURES

6.1 FIELD EQUIPMENT CALIBRATION

The reliability and credibility of field measurements will be ensured by calibration of sampling equipment. The following subsections review calibration procedures for field sampling equipment. Records of field equipment calibration will be maintained using the GAI Equipment Calibration Record form (Figure 4).

6.1.1 Air Monitoring

All air sampling equipment will be calibrated before and after each use or as otherwise directed by the sampling and analytical method or the equipment manufacturer. Where required, equipment will be calibrated each day during field use. The equipment manufacturer's calibration procedures will be followed. Calibration procedures for the following equipment to be used in the air monitoring study or for personnel air monitoring are summarized in Appendix D:

- TEI Model 43A Pulsed Fluorescence SO_2 Analyzer
- TEI Model 45 Pulsed Fluorescence H_2S Analyzer
- National Draeger Model 190 Data-Logger Gas Monitor
- HNu Model PI-101 Photoionization Detector
- MIE Model PDM-3 Mini-Ram Aerosol Monitor

7.0 ANALYTICAL PROCEDURES

7.1 AIR MONITORING

Samples collected to determine airborne benzene concentrations will be analyzed by Lancaster Laboratories, Inc., Lancaster, Pennsylvania, using the standard analytical methods detailed in Table 2. The methods to be used for the analysis of benzene is provided in Appendix B-1. The inorganic gases H_2S and SO_2 will be measured using direct-reading monitors, as detailed in Section 4, and do not require laboratory analyses. Detection limits for each sampling and analytical method are shown in Table 2.

Lancaster Laboratories, Inc., is accredited and certified by the American Industrial Hygiene Association and Pennsylvania Department of Environmental Resources, and is also in the EPA Contract Lab Program.

Specific details concerning analytical QA/QC procedures for Lancaster Laboratories, Inc. is presented in Appendix E.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

8.1 RECORDKEEPING

Recordkeeping systems have been implemented by HRD to provide adequate checks on site operation. The types of records include:

- Field data and testing records
- QA/QC records
- Inspection records
- Safety records
- Training records

The format of the records may change over the life of the operation due to regulatory changes and improvements in inspection and recordkeeping at HRD. The records systems and reports utilized will comply with the Consent Decree requirements and ensure the safe and proper operation of the Cinder Bank Operable Unit. These records will be maintained at HRD for a period of 6 years and will be properly classified and filed in chronological order for convenience of retrieval.

8.1.1 Testing Records

Various testing for air contaminants will be performed during the completion of this work. The detailed records and field data related to this testing will be compiled and maintained in the HRD project files.

8.1.2 Quality Assurance/Quality Control Records

Records relating to project QA/QC activities will be prepared and maintained in the project files. These records will typically include:

- Analytical QA (instrument calibration, internal blanks, etc.). These records will be maintained at the individual laboratories.
- Field sampling equipment calibration records.
- Analytical and field QA/QC data (sample blanks, replicates, field blanks).
- QA Manager QA/QC records. (This individual is responsible for implementing the QA/QC plan.

8.1.3 Other Records

Other records to be prepared, compiled, and maintained during the course of the project relate to the operation of the site. While these are not directly related to QA/QC, they do represent part of the project documentation. These will include:

- Site visitors log
- Inspection records
- Maintenance records
- Safety records
- Employee training and medical certification records

8.2 FIELD AND TECHNICAL DATA

The field and technical (nonlaboratory) data that will be collected can generally be characterized as either "objective" or "subjective" data.

Objective data include all direct measurements of field data, such as field screening/analytical parameters. Subjective data include descriptions and observations.

All data collection activities performed at a site will be documented either in the bound, numbered field log or on appropriate forms. Entries will be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory. All field log entries will be dated.

8.2.1 Data Reduction

After checking the data in the field notes, the lead Field Technician will reduce the data to tabular form, wherever possible, by entering the material in data files. Where appropriate, the data files will be set up for direct input into a database. Other objective data may be set up in spreadsheet-type tabular files (e.g., direct-reading air monitors). Subjective data will be filed as hard copies for later review and incorporation into technical reports, as appropriate.

8.2.2 Data Validation (Quality Control Procedure)

Validation of objective field and technical data will be performed at two different levels. On the first level, data will be validated at the time of collection by following standard procedures and QC checks. At the second level, data will be validated by the Project Manager, who will review the data to ensure that the correct codes and units have been included.

After data reduction into tables or arrays, the GAI Project Manager will review data sets for anomalous values. Any inconsistencies or anomalies discovered will be resolved immediately, if possible, by seeking clarification from the field personnel responsible for collecting the data.

Subjective field and technical data will be validated by the HRD Project Manager, who will review field reports for reasonableness and completeness. In addition, random checks of sampling and field conditions will be made by the GAI Project Manager, who will check recorded data at that time to confirm the recorded observations. Whenever possible, peer review will also be incorporated into the data validation process, particularly for subjective data, in order to maximize consistency between field personnel.

8.3 OFF-SITE LABORATORY DATA PROCEDURES

8.3.1 Data Logging

The sample custodian, upon receipt of samples for analyses accompanied by a completed request for analyses and/or Chain-of-Custody Record, will do the following:

- Verify completeness of submitted documents, including the Chain-of-Custody Records.
- Log in samples, assign unique lot numbers, and attach the number to the sample container(s).
- Open the project file and enter data in the laboratory tracking system.
- Store samples in refrigerated sample bank.

gai

8.3.2 Data Collection

In addition to the data collected in the field and recorded on the Chain-of-Custody Records, data describing the processing of samples will be accumulated in the laboratory and recorded in laboratory notebooks. Laboratory notebooks will contain the following:

- Date of processing.
- Sample numbers.
- Analyses or operation performed.
- Calibration data.
- Quality control samples included.
- Concentrations/dilutions required.
- Instrument readings.
- Special observations (optional).
- Analyst's signature.

8.3.3 Data Reduction

Data reduction is performed by the individual analysts and consists of calculating concentrations in samples from the raw data obtained from the measuring instruments. The complexity of the data reduction will be dependent on the specific analytical method and the number of discrete operations (extractions, dilutions, and concentrations) involved in obtaining a sample that can be measured.

For those methods using a calibration curve, sample response will be applied to the linear regression line to obtain an initial raw result, which is then factored into equations to obtain the

estimate of the concentration in the original sample. Rounding will not be performed until after the final result is obtained to minimize rounding errors, and results will not normally be expressed in more than two significant figures.

8.3.4 Data Review (Internal Quality Control)

System reviews are performed at all levels. The individual analyst constantly reviews the quality of data through calibration checks, quality control sample results, and performance evaluation samples. These reviews are performed prior to submission to the Laboratory Manager.

The Laboratory Manager will review data for the consistency and reasonableness with other generated data and determine if program requirements have been satisfied. Selected hard copy output of data (chromatograms, spectra, etc.) will be reviewed to ensure the results are interpreted correctly. Unusual or unexpected results will be reviewed, and a resolution will be made as to whether the analysis should be repeated. In addition, the Laboratory Manager will recalculate selected results to verify the calculation procedure.

The QA Manager independently conducts a complete review of selected projects to determine if laboratory and client quality assurance/quality control requirements have been met. Discrepancies will be reported to the appropriate Section Manager and/or Analytical Project Manager for resolution.

8.3.5 Data Reporting

Copies of all raw data and the calculations used to generate the final results will be compiled into a data documentation package to allow reconstruction of the data reduction process by a third party data validator. The data package will include all raw data, all instrument and/or equipment calibration results, calculations, blank results, duplicate results, Chain-of-Custody Records, and packing lists and/or air bills, and copies of analyst's logbooks (signed by the analyst and reviewed by a second party) with date and time of sample preparation and analysis.

A cover page summarizing chronology will be prepared for submittal with the package which includes: date received, client sample ID, laboratory sample ID, matrix, preparation batch reference, collection date, preparation date, and analysis date for all samples (including dilution and reruns) and corresponding QC samples.

The cover page and all sample report forms must be labeled with the complete client sample ID number as it appears on the Chain-of-Custody Record.

The case narrative must document all problems encountered and the subsequent resolutions. Instrumentation and methods employed for analysis must also be included. Note whether samples were preserved or not and the procedure utilized in preservation.

8.3.6 Laboratory Data Archiving

The laboratories will maintain on file all of the raw data, laboratory notebooks, and other documentation pertinent to the work on a given project. This file will be maintained by the laboratory during the duration of the project and for at least 16 years after its termination.

Data retrieval from archives will be handled in a similar fashion as a request for analysis. Specifically, a written work request to include a quotation must be submitted for retrieval of data. Client confidentiality will be maintained with retrieved data. Consequently, the laboratory can honor only those requests for data authorized by the original client.

8.4 DATA VALIDATION/USABILITY REVIEW (QUALITY ASSURANCE PROCEDURE)

Separate from the laboratory's internal data review/data validation, a review of the final data package will be performed to validate results and to determine usability. Criteria to assess usability will be taken from EPA's Functional Guidelines on Data Validation. The depth of review will depend on the data deliverable package. Guideline criteria will be applied to available documentation.

This validation will be performed at the direction of EPA by personnel other than those involved with the analysis.

8.5 DATA ARCHIVING

At the conclusion of this study, all the files for this project will be placed in "dead storage" at HRD's office. Prior to this time, the files will be active and open. The files will be preserved during the remediation and for at least six years after its termination.

Copies of all raw data and the calculations used to generate the final results will be compiled into a data documentation package to allow reconstruction of the data reduction process by a third party data validator. The data package will include all raw data, all instrument and/or equipment calibration results, calculations, blank results, duplicate results. Chain-of-Custody

Records, any packaging lists and/or air bills, and copies of analyst's logbooks (signed by the analyst and reviewed by a second party) with date and time of sample preparation and analysis.

A cover page summarizing sample chronology will be prepared for submittal with the package, and it will include the date received, client sample ID, laboratory sample ID, matrix, preparation batch reference, collection date, preparation date, and analysis date for all samples (including dilutions and reruns) and corresponding QC samples.

The cover page and all sample report forms must be labeled with the complete client sample ID number as it appears on the Chain-of-Custody Record.

The case narrative must document all problems encountered and the subsequent resolutions. Instrumentation and methods employed for analyses must also be included. Note whether samples were preserved or not and the procedure utilized in preservation.

9.0 INTERNAL QUALITY ASSURANCE/QUALITY CONTROL

The purpose of QA/QC checks is to produce precise, accurate, and complete data (QC of the testing procedures).

9.1 INTERNAL QUALITY ASSURANCE CHECKS

The contract laboratory selected to perform organic and selected inorganic analyses of the air monitoring will be required to have in place a QA program that will ensure consistency and continuity of data and that will be documented in a quality assurance plan (lab-specific quality assurance program plan are appended to this plan). these procedures include:

- Instrument performance checks.
- Instrument calibration.
- Retrieval of documentation pertaining to instrument standards, samples, and data.
- Documentation of analytical methodology and QC methodology (QC methodology includes spiked samples, duplicate samples, blanks, and check standards for method accuracy and precision).
- Documentation of sample preservation and transport.

Comprehensive QA records will be maintained to provide evidence of the QA activities. The contract Laboratory Manager will be responsible for ensuring that QA records are properly filed and stored so that they can be readily retrieved. QA criteria will be in conformance with SW-846 3rd edition requirements.

9.2 FIELD AND LABORATORY QUALITY CONTROL SAMPLES

All analytical work performed will comply with the QA/QC and analytical criteria specified in the test methods detailed in Sections 4 and 7.

Quality control checks to be instituted by field and laboratory personnel include:

- Field Blanks - A field equipment blank will be prepared for every five air samples and analyzed for all parameters.
- Laboratory Duplicates - The laboratory will perform a duplicate analysis of one sample for every 10 samples received.

10.0 PERFORMANCE AND SYSTEM AUDITS

10.1 GENERAL

Independent audits of on-site activities and field sampling procedures conducted by HRD representatives are anticipated during the course of the project. Audits, if any, will be conducted during actual field operations.

After such an audit has taken place, the author will be requested to brief the Site Manager or the On-Site Foreman to discuss any nonconforming actions or procedures observed. Corrective action (if any) that may be taken as a result of the audit will be documented in the project files.

10.2 FIELD AUDITS (QUALITY ASSURANCE PROCEDURE)

An unannounced audit of the site pertaining to conformance with QA/QC procedures may be performed by designated HRD personnel. The auditing of field operations is primarily performed for internal use. The auditor informs the HRD Project Manager of the audit the day prior to auditing. A written report on the results of this audit (and where necessary a notice of nonconformance) will be submitted to the following:

- EPA Site Manager
- HRD Project Manager
- HRD On-Site Foreman

A nonconformance notice describes any nonconforming conditions and sets a date for response and corrective action. The response is reviewed by the EPA Site Manager and, if satisfactory, is approved in writing.

gai

10.3 EXTERNAL LABORATORY AUDITS (QUALITY ASSURANCE PROCEDURE)

Unannounced audits of the laboratory may be conducted by EPA. Written reports on the results of these audits will be distributed to the same individuals listed in Subsection 10.2. Nonconformances will be addressed in a manner similar to the procedures applicable to field audits.

External audits are periodically conducted as requirements for formal laboratory certification programs.

11.0 PREVENTIVE MAINTENANCE

11.1 GENERAL EQUIPMENT MAINTENANCE AND REPAIR

Instruments will be maintained in accordance with manufacturer's specifications. More frequent maintenance may be required depending on operations performance. Instrument logs will be maintained to document the date and type of maintenance performed.

11.2 LABORATORY EQUIPMENT

Procedures for maintenance and calibration are in accordance with the manufacturer's specifications and are described in the Laboratory's Quality Assurance Plan.

12.0 DATA ASSESSMENT PROCEDURES

12.1 INTRODUCTION

The QA objectives for precision, accuracy, representativeness, completeness, and comparability were given and discussed in Section 3. All analytical data are reviewed relative to these criteria and specific project requirements to assess the quality of the analytical data. Adherence to the representativeness and comparability goals will allow evaluations of the analyzed samples to be extrapolated to the rest of the project. Where all criteria are met, data are deemed acceptable without qualification. Where precision and accuracy goals are not met, the sample set is reanalyzed or reported with qualification. Some of the factors affecting this final sample disposition include:

- Project-specific QA/QC requirements.
- Availability of sufficient sample for re-analysis.
- Regulatory action limits.

12.2 PRECISION

Precision is measured through analyses of replicate QC control and field samples. Results from these standards are calculated as relative percent difference (% RPD) or percent relative standard deviation (% RSD).

Precision measurements from field samples give an indication of sample homogeneity. Problems with sample homogeneity are more likely to occur with Ecoloam™ and Cinder Bank material samples.

12.3 ACCURACY

Accuracy is measured through the analysis of fortified reagent free matrices and fortified field samples. Results from these measurements are calculated as percent recovery.

Accuracy measurements from field samples give an indication of physical or chemical interferences present which can either enhance or mask the actual presence of target analytes. Determination of percent recovery (% R) requires analysis of a fortified sample and a nonfortified sample, so that any background analyte already present in the sample can be accounted for in the recovery determination. Thus, sample homogeneity also becomes a factor in recovery determinations, as variable background can affect the apparent analyte recovery.

12.4 COMPLETENESS

Completeness is a measure of the amount of analytical data gathered by an analytical method or system meeting all accuracy and precision criteria. The minimum goal for completeness is 80% and the ability to exceed this goal is dependent on the applicability of the analytical methods to the sample matrices analyzed. However, even if data have not met this laboratory definition of data able to be reported without qualification, project completion goals may still be met if the qualified data, i.e, data of known quality even if not perfect, is suitable for specified project goals.

13.0 FEEDBACK AND CORRECTIVE ACTION

13.1 OVERVIEW

As part of the overall QA process, when a nonconformance or deficiency is identified during a formal audit or during a routine QC audit of field activities, corrective action will be initiated by the Site Manager. The Site Manager will also be responsible for ensuring that the correction action has actually been taken, and that it adequately addresses the nonconformance. When corrective actions are required in an on-site or off-site laboratory, the appropriate Laboratory Manager will determine appropriate corrective actions in that off-site laboratory.

13.2 NONLABORATORY ACTIVITIES

Participating staff will be responsible for reporting suspected QA nonconformance by initiating nonconformances that are implemented by the following actions:

- Evaluating reported nonconformances.
- Controlling additional work on nonconformance items.
- Determining disposition of action to be taken.
- Maintaining a log of nonconformances.
- Reviewing nonconformance reports.
- Evaluating disposition or action taken.
- Ensuring that nonconformance reports are included in the final site documentation.

The On-Site Coordinator (Foreman) will ensure that additional work that is dependent on the nonconforming activity is not performed until the nonconformance is corrected and will be responsible for evaluating each nonconformance and implementing corrective actions.

13.3 LABORATORY ACTIVITIES

The initial responsibility to monitor the quality of an analytical system lies with the analyst. In this pursuit, the analyst will verify that all QC procedures are followed and results of analysis of QC samples are within acceptance criteria. This requires that the analyst assess the correctness of all the following items as appropriate:

- Sample preparation
- Initial calibration
- Calibration verification
- Method blank result
- Duplicate analysis
- Laboratory control standard
- Fortified sample result

If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. The analyst notifies the appropriate supervisor and the QA Section Manager of the problem and, if possible, identifies potential causes and corrective action.

The nature of the corrective action obviously depends on the nature of the problem. For example, if a continuing calibration verification is determined to be out of control, the corrective

action may require recalibration of the analytical system and reanalysis of all samples since the last acceptable continuing calibration standard.

When the appropriate corrective action measures have been defined and the analytical system is determined to be "in control", the analyst documents the problem, and the corrective action. Copies of the form summarizing these actions are provided to the Section Manager and QA Section Manager.

Data generated concurrently with an out-of-control system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency noted in the case narrative. Where sample results are impaired, the Laboratory Manager is notified and appropriate corrective action (e.g. reanalysis) is taken.

The critical path for assessing the correctness and acceptability of analytical data is shown in Figure 4.

14.0 QUALITY ASSURANCE REPORTS AND OPERATION REPORTS

14.1 RESPONSIBILITY

The proper maintenance of QA records is essential to ensure the overall quality of the investigation and is part of the overall QA Process. Comprehensive QC records will be maintained in the project file to provide evidence of the QA activities. Records of QC program implementation will be written and retained on file. QA documents will be archived in the project files. Pertinent information, including that received from subcontractors and other outside sources or developed during the project, will be maintained.

The QA Manager will compile information relative to QA objectives, audits, nonconformance reports, and any significant QC deficiencies. The QA Manager will be responsible for ensuring that QC records are properly filed and stored. Access to all project files will be controlled by the QA Manager. QA reports to management will also be part of the project file and will include quarterly QC reports, audit report, and corrective action results.

Refer to Section 14 of the Blue Mountain QAPP for additional detail of these requirements.

15.0 REFERENCES

1. U.S. Environmental Protection Agency (U.S. EPA), "Record of Decision, Palmerton Zinc Site Operable Unit II, Cinder Bank," June 29, 1988.

2. U.S. EPA, "Administrative Order by Consent, Palmerton Zinc Site Cinder Bank," September 1985.

3. U.S. EPA, "Amendment to Administrative Order by Consent, Palmerton Zinc Site Cinder Bank," December 13, 1991.

4. GAI Consultants, Inc., "Air Monitoring Program at Cinder Bank Operable Unit (Operable Unit 2 - Palmerton Zinc NPL Site)," April 1993.

5. Zinc Corporation of America, "Quality Assurance Project Plan for the Blue Mountain Operable Unit (Palmerton Zinc NPL Site) (Appendix 2 of the Remedial Design)," April 1991.

6. GAI Consultants, Inc., "Quality Assurance Plan for Additional Studies for Remedial Action at Cinder Bank Operable Unit (Operable Unit 2 - Palmerton Zinc NPL Site)," May 1992.

7. U.S. EPA, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," EPA-600/4-83-004, February 1983.

8. GAI Consultants, Inc., "Work Plan for Additional Studies for Remedial Action at Cinder Bank Superfund Site, Supplemental Air Monitoring," May 1994.

9. GAI Consultants, Inc., "Site Health and Safety Plan for Additional Studies for Remedial Action at Cinder Bank Operable Unit (Operable Unit 2 - Palmerton Zinc NPL Site)," May 1992.

92118qap.hat/cwi38

TABLES

Table 1

SUMMARY OF DATA NEEDS AND QUALITY OBJECTIVES

Study Objective	Data Needed	Proposed Quality Control Data Collection and Evaluation Activities	Analytical Level*
Assess potential release of air contaminants from venting areas.	Determine if potential air contaminants are present and estimate emission rates.	Perform air sampling for air contaminants detailed in Table 2.	II/III
Ensure the health and safety of on-site work and public in vicinity of cinder bank.	Determine if proper health and safety protocols are followed and if situations exist that may threaten health and safety of on-site and nearby personnel.	Perform air monitoring for dust and toxic gases during on-site activities, implement procedures and operational controls and inspections as required by the HASP.	I/II

Note:

* As defined in Table 4-3 of "Data Quality Objectives for Remedial Response Activities," U.S. EPA, EPA/540/G-87/003, March 1987.

- Level I - Field screening or analysis using portable instruments.
- Level II - Field analyses using more sophisticated portable analytical instruments.
- Level III - All analysis performed in an off-site analytical laboratory.

Table 2

QUALITY ASSURANCE OBJECTIVES
AND METHOD SUMMARY

SUPPLEMENTAL AIR MONITORING STUDY

Contaminant	Sampling and Analytical Method	Target Air Concentration	Required Sample Time	Optimal Sample Volume	Minimum Quantifiable Concentration	Method Precision
Sulfur Dioxide	EQSA-0486-060	80 $\mu g/m^3$ (0.03 ppm)	5 minutes	N/A	0.4 ppb	1%
Hydrogen Sulfide	TEI 45B	0.94 $\mu g/m^3$ (0.7 ppb)	5 minutes	N/A	0.6 ppb	1%
Benzene	TO-1	0.29 $\mu g/m^3$ (0.09 ppb)	N/A	N/A	0.1 ppb	20%

Note:

N/A - Not Available

Table 3

SUMMARY OF PERSONAL AIR MONITORING
TO BE CONDUCTED DURING
CINDER BANK SITE ACTIVITIES

Parameter to be Evaluated	Monitoring Method	OSHA PEL	Site Action Level
Total Particulate - Nuisance Dust	MIE Model PDM-3 Mini-Ram Aerosol Monitor	15 mg/m^3	5 mg/m^3 (as total dust)
Hydrogen Sulfide	National Draeger Model 190 Data-Logger Gas Monitor (with H_2S specific detector)	10 ppm - TWA 15 ppm - STEL	5 ppm
Sulfur Dioxide	National Draeger Model 190 Data-Logger Gas Monitor (with SO_2 specific detector)	2 ppm - TWA 5 ppm - STEL	1 ppm
Total Hydrocarbons	HNu Model PI-101 Photoionization Detector	---	50 ppm

ORIGINAL (Red)

Table 4

SAMPLE IDENTIFICATION CODES

Project Task	Sample Type	Sample Identification Code
Supplemental Air Monitoring	Direct-reading Instrument	92-118-10-DR-MMDDYY-XXX
	Air sample in SUMMA® canister	92-118-1-SU-MMDDYY-XXX

Notes:

MM - Calendar month (i.e., 01 = January, 02 = February, etc.)

DD - Calendar day

YY - Calendar year

XXX - Unique sample number, not to be repeated.

gai

FIGURES

FIGURE 1

IMPLEMENTATION SCHEDULE
SUPPLEMENTAL AIR MONITORING

HORSEHEAD RESOURCE DEVELOPMENT COMPANY, INC.

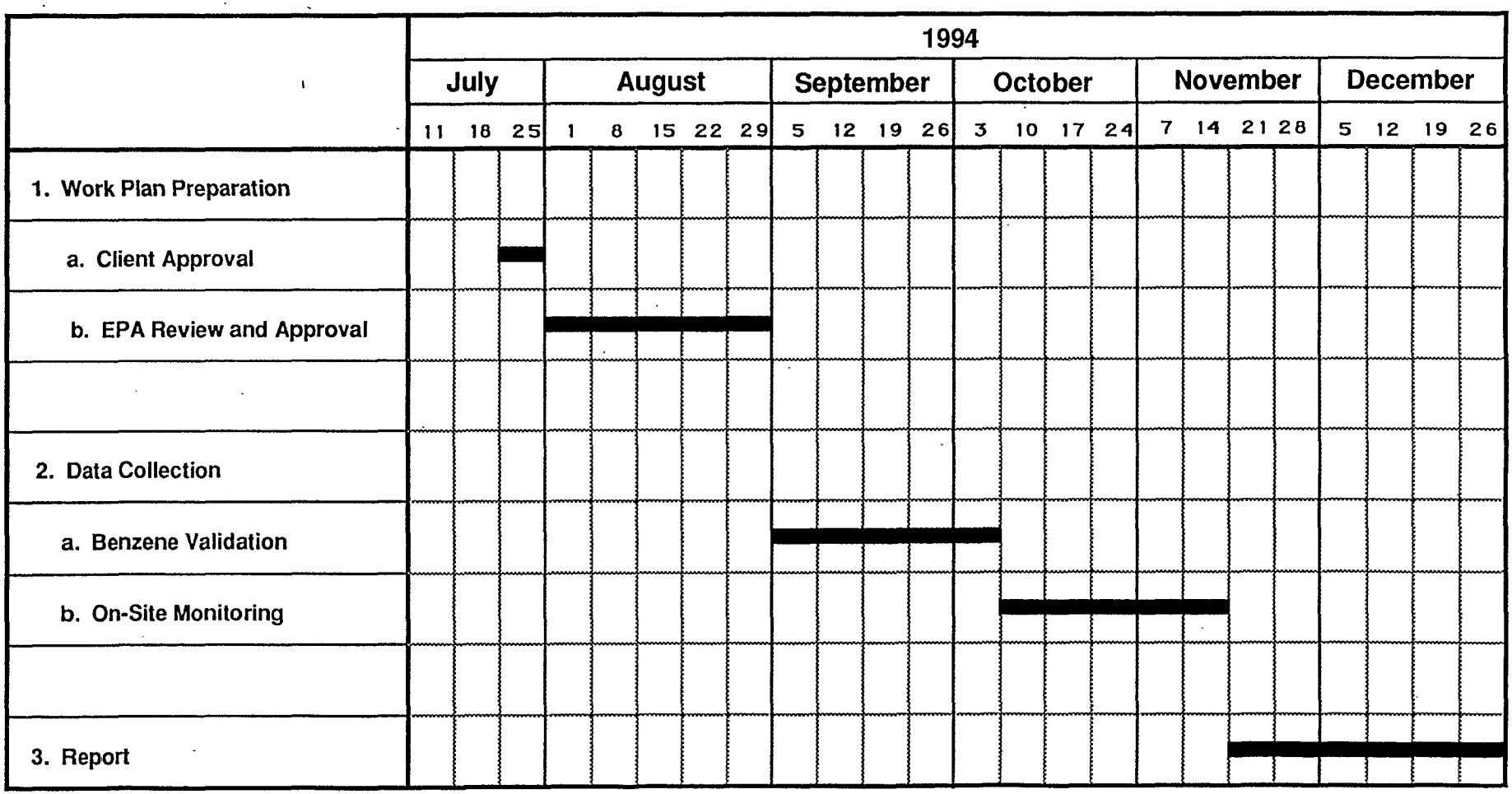
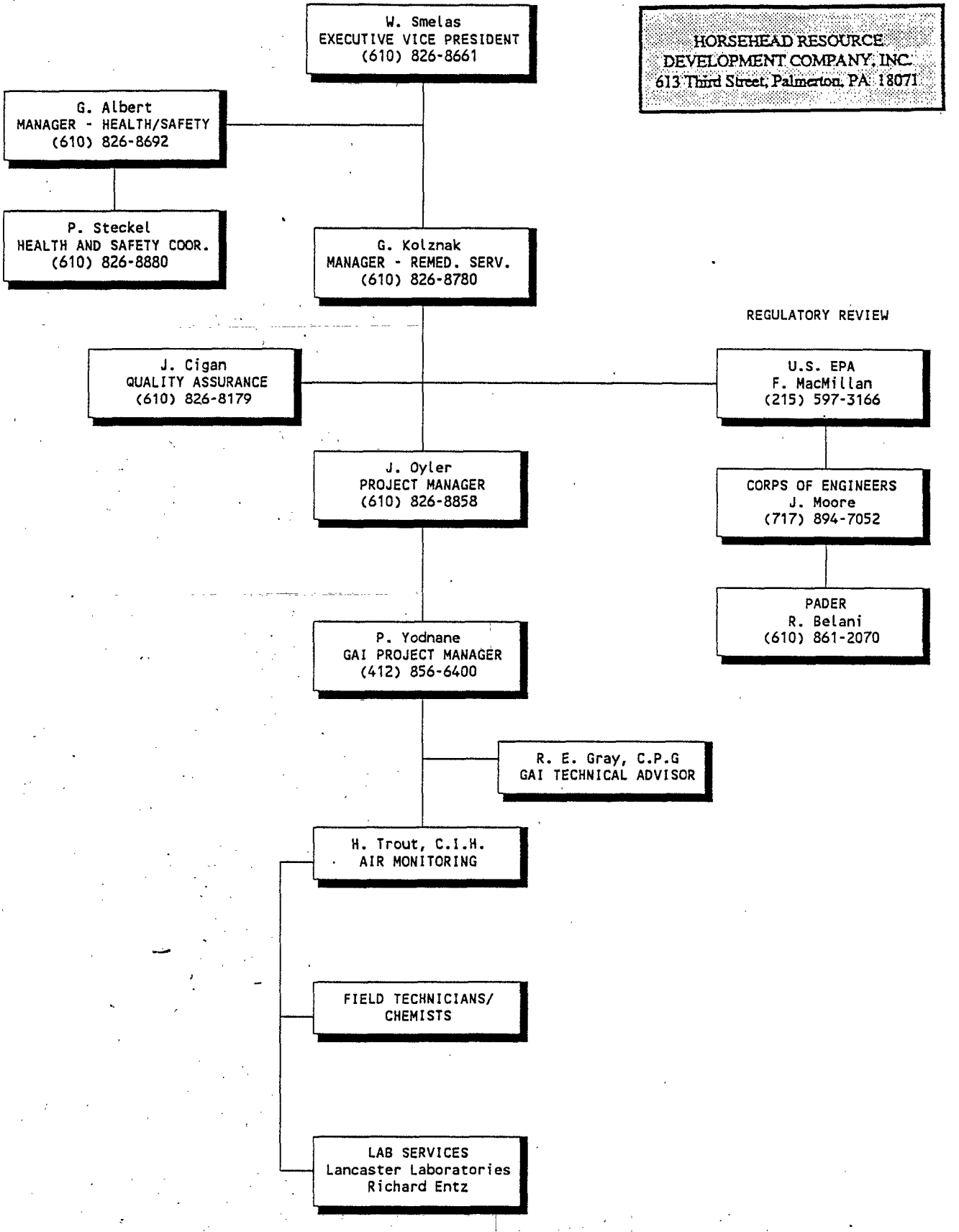


Figure 2
PROJECT ORGANIZATION CHART

ORIGINAL (Red)



AR303742

CHAIN OF CUSTODY RECORD

CONSULTANTS, INC.

PROJECT NO.	PROJECT NAME					NO. OF CON-TAINERS	ANALYSES								REMARKS
SAMPLERS: (SIGNATURE)															
STATION NUMBER AND LOCATION	SAMPLE MATRIX	DATE	TIME	COMP	GRAB										

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)
Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks	

AR303743

Figure 3

Calibration Record

PROJECT NUMBER _____

GAI CONSULTANTS, INC.

EQUIPMENT CALIBRATION RECORD

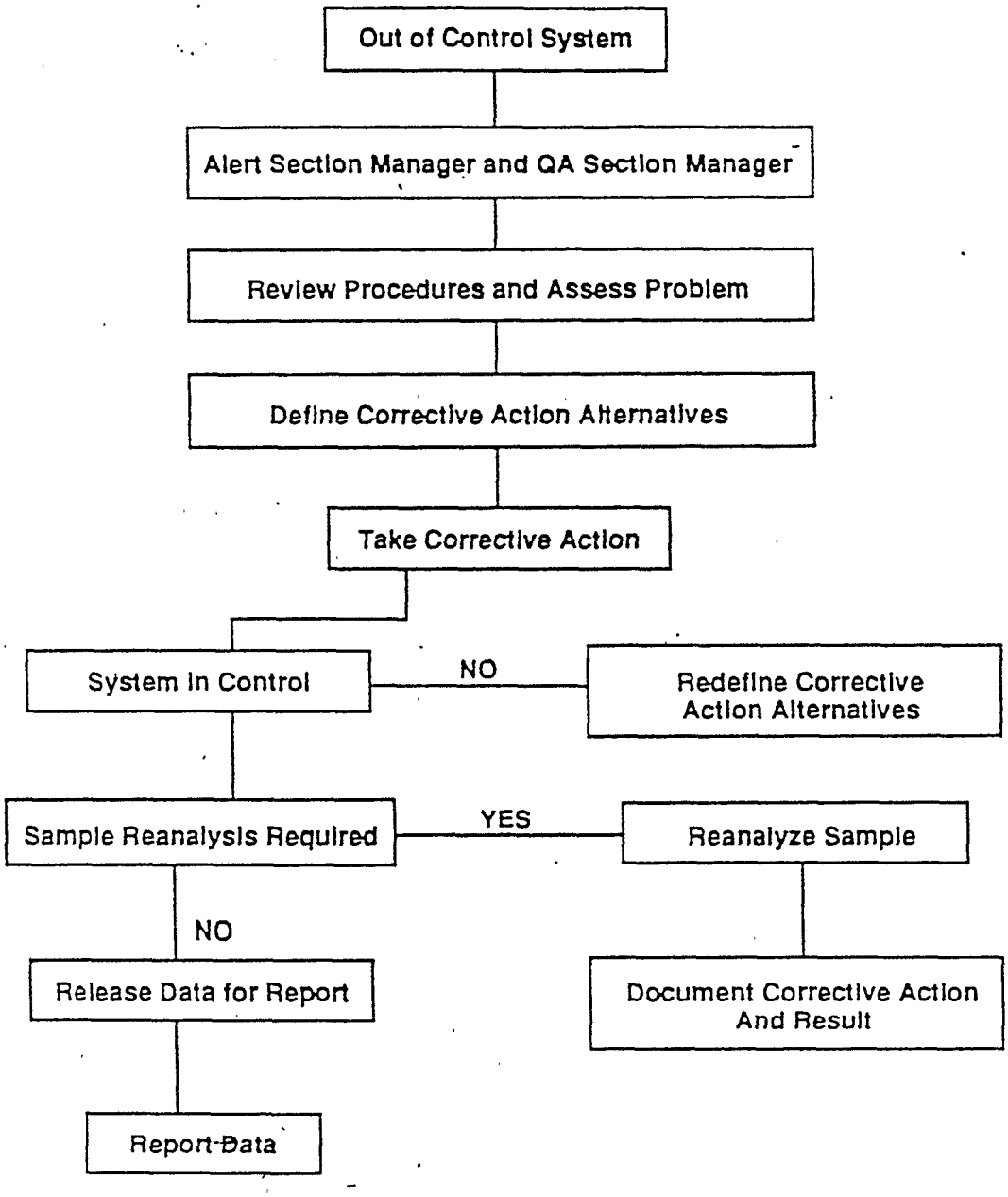
EQUIPMENT TYPE	DATE OF CALIBRATION	MANUFACTURER AND MODEL NUMBER	SERIAL NUMBER	STANDARD UTILIZED / MATRIX	SPAN SETTING	CALIBRATION PERFORMED BY	REMARKS

AR303744

copy to Laboratory QA Manager, Project Manager

Figure 4

ORIGINAL (Red)



CRITICAL PATH FOR CORRECTIVE ACTION

Figure 5

APPENDIX A

RESUMES OF PERTINENT PROJECT PERSONNEL

HRD EMPLOYEE PROFILES

William A. Smelas

William A. Smelas is Executive Vice President of HRD and President of the Metals Recovery Division. Bill joined the New Jersey Zinc Company in 1960 and is a Chemical Engineering Graduate from the University of Pittsburgh. He has held various operating and management positions for NJZ and HRD.

George A. Kolznak

George A. Kolznak is Manager of Remediation Services for HRD. Mr. Kolznak was employed by the New Jersey Zinc Company for 12 years in numerous positions both in Research and Operations. Prior to joining HRD in 1991, he had an extensive background in construction and plant management. He holds a B.S. in Chemistry from Wayne State University.

John A. Oyler

John Oyler is a Reclamation Scientist in the Remediation Services Division of HRD and is Project Manager for the Superfund activities in Palmerton. Prior to joining HRD in August 1987, he had extensive background with the USDA Soil Conservation Service in rectifying environmental problems. He earned a B.S. degree in Forestry with specialization in forest ecology from Southern Illinois University and has authored numerous articles and papers published in environmental and agronomy journals.

John M. Cigan

John Cigan joined HRD in April 1989 as the Director of Technical Services with overall responsibility for the quality control/quality assurance compliance, analytical services and industrial hygiene functions. He holds a Ph.D. in metallurgical engineering from Carnegie-Mellon University and has 25 years of similar management and technical experience prior to joining HRD.

George R. Albert

George R. Albert is employed by HRD as Manager - Health and Safety. He has 21 years with the Company in numerous positions in operations and health and safety. He presently oversees the Health and Safety program for all of HRD (all locations) and has conducted the program at Palmerton since May, 1989. He has an A.A. degree in accounting from Lehigh County Community College.

Paul H. Steckel

Paul Steckel was employed by HRD on 16 October, 1990 as the Director of Environmental Compliance Services with support responsibilities at HRD and ZCA operations. He functions as the Site Health and Safety Coordinator for the Superfund activity on the Blue Mountain and Cinder Bank operable units. He previously held management positions at Lone Star Steel and U.S. Steel, and was an Associate Attorney in a private law practice. He holds a B.S. in Metallurgical Engineering from Lafayette College and a J.D. from Dickinson School of Law.

HARRY A. TROUT

Director of Health and Safety

AREAS OF SPECIALIZATION

Industrial hygiene, occupational health and safety, hazardous materials control, chemical safety, environmental and public health risk assessment. Site assessment for the evaluation, control, and remediation of hazardous materials and waste, asbestos, PCBs, and air contaminants.

EDUCATIONAL BACKGROUND

B.S. Chemistry 1977
Saint Vincent College

M.S. Industrial Hygiene 1979
University of Pittsburgh

PROFESSIONAL CERTIFICATIONS

Certified Industrial Hygienist
Certified Safety Professional
Certified Asbestos Hazard Evaluation Specialist, Ohio
Certified Asbestos Inspector and Management Planner, Pennsylvania and West Virginia
Previously Certified AHERA Asbestos Project Designer and Supervisor

EMPLOYMENT HISTORY

1991-present	GAI Consultants
1988-1991	Libbey-Owens-Ford Co.
1985-1988	Schneider Engineers
1982-1985	The Mead Corporation
1979-1982	*Koppers Company, Inc.*

PROFESSIONAL EXPERIENCE

- Analysis of noise in industrial and community locations to evaluate potential impact of exposure on employees and sensitive receptors in the community; design and installation of noise control and source reduction techniques; and development of audiometric testing and hearing conservation programs.
- Management of safety, health, and loss control programs for a major producer of flat glass products serving the automotive and architectural markets. Development of a company-wide drug and alcohol program, motor vehicle safety program, and medical case management systems.
- Management and administration of the Industrial Hygiene and Environmental Health Group in an ENR 50 engineering firm. Comprehensive industrial hygiene consultation services, technical supervision, personnel and financial management, and planning and marketing of services. General industrial hygiene surveys, asbestos hazard assessment and abatement, hazard communication and right-to-know, PCB hazard evaluation and disposal, and hazardous materials and waste management.
- Preparation and administration of the Quality Assurance Project Plan and the Health and Safety Plan for remedial action studies completed at a Superfund site in eastern Pennsylvania.
- Industrial hygiene surveys for chemical, noise, heat, radiation, and biological exposure, including indoor air quality evaluations, OSHA compliance evaluations, and design of control methods. Development of chemical control, hazard communication, hearing conservation, medical surveillance, ergonomics, asbestos management, personal protective equipment, and exposure monitoring programs.
- Project manager or senior technical consultant for asbestos hazard assessment and management projects, including initial assessment and sampling, remediation project design, specification preparation, and project management and oversight during abatement for public sector clients in the manufacturing, utility, and real estate management industries.
- *Project manager/technical consultant for environmental* remediation activities involving asbestos, PCBs, lead-based paint, and other hazardous materials for the renovation of the 1.9 millon sq ft John Wanamaker Building and the 1 millon sq ft Lit Brothers Building in Philadelphia, PA.
- Project technical consultant for more than 50 comprehensive real estate environmental assessments of industrial and commercial properties conducted as part of acquisition or divestiture by the client. Site inspection, sampling and analysis, risk assessment, remediation design, and contractor oversight.
- Management of safety, health, loss prevention, product safety, and environmental concerns for the start-up division of a large corporation. Development of a division safety program in a research and development atmosphere and a product safety and toxicology program to support the development of a proprietary non-photographic imaging system.
- Development and administration of corporate-wide industrial hygiene and occupational health programs for a Fortune 150 forest products company. Development and implementation of chemical hazard communication, asbestos abatement, and hearing conservation programs and management of the corporation's industrial hygiene and product safety laboratory.
- Preparation and administration of site-specific health and safety plans for field investigation and remediation at uncontrolled waste sites.
- Design and completion of Superfund risk assessments.

PROFESSIONAL AFFILIATIONS

American Industrial Hygiene Association
American Society of Safety Engineers
American Chemical Society
National Fire Protection Association
Air and Waste Management Association
Board of Certified Safety Professionals
American Board of Industrial Hygiene

gai

PRECHA YODNANE

Engineering Manager

AREAS OF SPECIALIZATION

Environmental engineering for industrial projects. Solid and hazardous waste management and permitting, remedial investigation and remedial design of Superfund sites, regulatory reviews and environmental compliance programs, conceptual design for process plant pollution control systems, water and wastewater treatment, RCRA and NPDES permitting, spill prevention and control, air pollution permitting, industrial siting studies, noise modeling and control, and radiation safety programs.

PROFESSIONAL REGISTRATION

Professional Engineer, Pennsylvania, Maryland, Indiana

PROFESSIONAL CERTIFICATION

OSHA 40-Hour Hazardous Waste Operations and Superfund Worker Protection

EDUCATIONAL BACKGROUND

B.S. Sanitary Engineering 1972
Bangkok, Thailand

M.S. Civil Engineering 1975
University of Pittsburgh

Ph.D. Civil Engineering 1978
University of Pittsburgh

EMPLOYMENT HISTORY

1984-present	GAI Consultants
1983-1984	*Biothane Corporation*
1980-1983	Air Products & Chemicals, Inc.
1978-1980	Ashland Synthetic Fuels, Inc.
1975-1978	University of Pittsburgh

PROFESSIONAL EXPERIENCE

- Project manager for statewide open-end hazardous waste management services for the Pennsylvania Department of Transportation. Assignments included an environmental site assessment, waste site investigations, waste management plan preparation, site remediation and design, and waste management training. Eighteen projects have been assigned.
- Project manager for an open-end environmental contract for the Pennsylvania Turnpike Commission. Project services included emergency spill response and control, spill release investigations, corrective action plans, disposal of substances, closure reports for underground storage tanks, water and wastewater treatment systems design, and NPDES permitting. Twenty projects have been assigned.
- Project manager for additional studies for remedial action at the Cinder Bank Superfund Site, Palmerton, PA. Study included an air monitoring program, a site cover and cap evaluation, delineation of the burning area, and location of fire trenches.
- Project manager for the preliminary design of a power plant scrubber sludge landfill and a leachate treatment plant.
- Project engineer for the evaluation and design of a wastewater treatment system for landfills for hazardous waste and residual waste generated by steel manufacturing facilities.
- Project manager for the preparation of an operation and maintenance manual for the California Gulch Superfund Site wastewater treatment facility, Leadville, CO, and East Helena Superfund Site, MT.
- Project manager for a wastewater treatment system pilot study utilizing wetlands at the Blue Mountain Superfund Site, Palmerton, PA.
- Project engineer for treatability studies and facility design of wastewater treatment systems for several power generating stations, ash disposal sites, flue gas desulfurization facilities, and industrial discharges.
- Project manager/project engineer for site assessment, remediation assessment, remedial design for corrective action plans for contaminated soil and ground water at numerous industrial facilities and hazardous waste disposal sites.
- Project manager/project engineer for the treatability study, conceptual design, and specification of the treatment process for ground water collected from the Chisman Creek Superfund Site in Virginia, the Blue Mountain Superfund Site in Pennsylvania, and several other industrial facilities.
- Project engineer for the preparation of oil discharge contingency plans for thirteen Virginia Power facilities.
- Project engineer for remedial designs, including sludge stabilization/bedrock neutralization, emergency gases release modeling, and wastewater treatment, for Bruin Lagoon Superfund Site in Pennsylvania.
- Project manager/project engineer for feasibility studies and designs for the closure and post-closure of hazardous waste treatment, storage, and disposal facilities.
- Project engineer for evaluation of alternative water supply options for Virginia Electric and Power Company's Portsmouth Power Station ash sluicing system.
- Project manager/project engineer for delisting studies and the preparation of Part A and Part B RCRA permit applications for hazardous waste disposal facilities in Butler, PA, and in Belpre, OH.
- Project engineer for the preparation of a flow and water quality monitoring program report as well as a surface water sampling manual for various waste disposal sites and power generating plants in Pennsylvania, West Virginia, and Maryland.
- Project manager for the water supply system replacement at Elkem Metal Company, Marietta, OH.
- Project manager for a sewage treatment system evaluation at the USAir Training Center.
- Project engineer for the upgrading of seven sewage treatment facilities at various U.S. Army installations in the Pittsburgh area.
- Project engineer for the development of an industrial pretreatment program for Johnstown, PA, and several sewage treatment facilities.

- Project engineer responsible for the air, noise, and energy analysis and noise barrier design for the environmental impact assessment for natural gas compressor stations and highway projects.

- Senior environmental engineer responsible for the conceptual design for process plant environmental control systems, the preparation of environmental reports, and computer-simulated air emissions for permit applications. Wastewater, cooling water, and boiler water treatment for industrial gas facilities and chemical plants; pure oxygen-activated sludge system performance tests; and review of a wastewater treatment plant design for a coal liquefaction plant (SRC-1).

- Senior process engineer responsible for the process design, mechanical design, and site layout for a proprietary anaerobic wastewater treatment system. Design of gas handling and safety systems for gas produced by the treatment plant. Management of the project and preparation of cost estimates and proposals.

- Environmental engineer in a coal liquefaction plant involved with wastewater treatment plant design, start-up, and operations; treatability studies of coal liquefaction wastewater and solvent; extraction of phenol from coal liquefaction wastewater; solid and hazardous waste disposal; air pollution control; noise survey and control; and annual and quarterly reports of environmental permits.

PROFESSIONAL AFFILIATIONS

American Society of Civil Engineers
Air and Waste Management Association
Water Pollution Control Federation

PUBLICATIONS

Yodnane, P., Okorn, D. W., and Marshall, B. M. "Evaluation of Dewatering and Treatment System at the Chisman Creek Superfund Site." Presented at the 1992 National Conference on Environmental Engineering Water Forum 92, Baltimore, Maryland, August 1992.

Yodnane, P., Perry, M. T., and Shumway, L. C. "Noise Barrier Design Alternatives for a Proposed Natural Gas Compressor Station." Presented at the 85th Annual Conference of the Air and Waste Management Association, Kansas City, Missouri, June 1992.

Okorn, D. W., Yodnane, P., and Perry, M. T. "Site Characterization and Corrective Measures for an Underground Storage Tank Site." Presented at the 85th Annual Conference of the Air and Waste Management Association, Kansas City, Missouri, June 1992.

Yodnane, P., Patelunas, G. M., and Niece, J. E. "Removal of Arsenic and Selenium from Fly Ash Leachate Using Iron Coprecipitation and Activated Alumina Adsorption." Presented at the 64th Annual Conference of the Water Pollution Control Federation, Toronto, Ontario, October 6-10, 1991.

Yodnane, P. "Methodology and Scope of Work for Hazardous Waste Management Projects." Presented at the First Thai Professional in American Technology Transfer Cooperation Seminar, Bangkok, Thailand, August 18-21, 1991.

Yodnane, P., Shumway, L. C., and Niece, J. E. "Effect of Natural Gas Fired Compressor Stations on Community Noise Levels." Presented at the 84th Annual Conference of the Air and Waste Management Association, Vancouver, British Columbia, June 16-21, 1991.

Patelunas, G. M., Yodnane, P., Davis, R. F., and Niece, J. E. "Waste Compatibility Testing and Stabilization Mix Design for Bruin Lagoon Superfund Site." Presented at the Sixth National RCRA/Superfund Conference, New Orleans, Louisiana, April 12-14, 1989.

Yodnane, P., Okorn, D. W., and Williams, R. J. "Treatability Study and Design of a Ground-Water Treatment System at Chisman Creek Superfund Site." In *Proceedings of the International Conference on Physiochemical and Biological Detoxification of Hazardous Waste*, Atlantic City, New Jersey, May 1988.

Yodnane, P., Okorn, D. W., and Williams, R. J. "Treatability Study and Design of a Treatment Plant for Leachate Generated from a Coal Ash Disposal Site." In *Proceedings of the Eighteenth Mid-Atlantic Industrial Waste Conference*, Technomic Publishing Co., Inc., 1986.

Yodnane, P., and Neufeld, R. "Enhanced Wastewater Purification via the Addition of Granular Coals and Chars to Activated Sludge." *Journal of the Water Pollution Control Federation*, March 1978.

Yodnane, P., Neufeld, R., and Abrams. "Preliminary Evaluation of the Use of Powdered Coal as a Natural Gas Substitution for Sewage Sludge Incineration." Report prepared for the U.S. Department of Energy, June 1977.

Yodnane, P., and Neufeld, R. "Upgrading of Activated Sludge via the Addition of Coals and Coal Gasification Plant Chars." Presented at the Thirty-First Industrial Waste Conference, Engineering Bulletin, Purdue University, May 1976.

APPENDIX B

STANDARD SAMPLING AND ANALYTICAL METHODS

B-1

METHOD T014
DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCS) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

gai

METHOD TO14

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

1. Scope

1.1 This document describes a procedure for sampling and analysis of volatile organic compounds (VOCs) in ambient air. The method is based on collection of whole air samples in SUMMA® passivated stainless steel canisters. The VOCs are subsequently separated by gas chromatography and measured by mass-selective detector or multidetector techniques. This method presents procedures for sampling into canisters to final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampling).

1.2 This method is applicable to specific VOCs that have been tested and determined to be stable when stored in pressurized and subatmospheric pressure canisters. Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters (1,2). However, minimal documentation is currently available demonstrating stability of VOCs in subatmospheric pressure canisters.

1.3 The organic compounds that have been successfully collected in pressurized canisters by this method are listed in Table 1. These compounds have been successfully measured at the parts per billion by volume (ppbv) level.

2. Applicable Documents

2.1 ASTM Standards

D1356 - Definition of Terms Related to Atmospheric Sampling and Analysis
E260 - Recommended Practice for General Gas Chromatography Procedures
E355 - Practice for Gas Chromatography Terms and Relationships

2.2 Other Documents

U.S. Environmental Protection Agency Technical Assistance Document (3)
Laboratory and Ambient Air Studies (4-17)

Revision 1.0
June, 1988

METHOD TO14

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

OUTLINE

OUTLINE (Cont.)

3. Summary of Method

3.1 Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister and a pump-ventilated sample line during sample collection. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of ambient air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated SUMMA® passivated canister.

3.2 After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to a predetermined laboratory for analysis.

3.3 Upon receipt at the laboratory, the canister tag data is recorded and the canister is attached to the analytical system. During analysis, water vapor is reduced in the gas stream by a Nafion® dryer (if applicable), and the VOCs are then concentrated by collection in a cryogenically-cooled trap. The cryogen is then removed and the temperature of the trap is raised. The VOCs originally collected in the trap are revolatilized, separated on a GC column, then detected by one or more detectors for identification and quantitation.

3.4 The analytical strategy for Method TO14 involves using a high-resolution gas chromatograph (GC) coupled to one or more appropriate GC detectors. Historically, detectors for a GC have been divided into two groups: non-specific detectors and specific detectors. The non-specific detectors include, but are not limited to, the nitrogen-phosphorus detector (NPD), the flame ionization detector (FID), the electron capture detector (ECD) and the photoionization detector (PID). The specific detectors include the mass spectrometer (MS) operating in either the selected ion monitoring (SIM) mode or the SCAN mode, or the ion trap detector. The use of these detectors or a combination of these detectors as part of an analytical scheme is determined by the required specificity and sensitivity of the application. While the non-specific detectors are less expensive per analysis and in some cases more sensitive than the specific detector, they vary in specificity and sensitivity for a specific class of compounds. For instance, if multiple halogenated compounds are targeted,

an ECD is usually chosen; if only compounds containing nitrogen or phosphorus are of interest, a NPD can be used; or, if a variety of hydrocarbon compounds are sought, the broad response of the FID or PID is appropriate. In each of these cases, however, the specific identification of the compound within the class is determined only by its retention time, which can be subject to shifts or to interference from other nontargeted compounds. When misidentification occurs, the error is generally a result of a cluttered chromatogram, making peak assignment difficult. In particular, the more volatile organics (chloroethanes, ethyltoluenes, dichlorobenzenes, and various freons) exhibit less well defined chromatographic peaks, leading to misidentification using non-specific detectors. Quantitative comparisons indicate that the FID is more subject to error than the ECD because the ECD is a much more selective detector for a smaller class of compounds which exhibits a stronger response. Identification errors, however, can be reduced by: (a) employing simultaneous detection by different detectors or (b) correlating retention times from different GC columns for confirmation. In either case, interferences on the non-specific detectors can still cause error in identifying a complex sample. The non-specific detector system (GC-NPD-FID-ECD-PID), however, has been used for approximate quantitation of relatively clean samples. The nonspecific detector system can provide a "snapshot" of the constituents in the sample, allowing determination of:

- Extent of misidentification due to overlapping peaks,
- Position of the VOCs within or not within the concentration range of anticipated further analysis by specific detectors (GC-MS-SCAN-SIM) (if not, the sample is further diluted), and
- Existence of unexpected peaks which need further identification by specific detectors.

On the other hand, the use of specific detectors (MS coupled to a GC) allows positive compound identification, thus lending itself to more specificity than the multidetector GC. Operating in the SIM mode, the MS can readily approach the same sensitivity as the

multidetector system, but its flexibility is limited. For SIM operation, the MS is programmed to acquire data for a limited number of targeted compounds while disregarding other acquired information. In the SCAN mode, however, the MS becomes a universal detector, often detecting compounds which are not detected by the multidetector approach. The GC-MS-SCAN will provide positive identification, while the GC-MS-SIM procedure provides quantitation of a restricted "target compound" list of VOCs.

The analyst often must decide whether to use specific or non-specific detectors by considering such factors as project objectives, desired detection limits, equipment availability, cost and personnel capability in developing an analytical strategy. A list of some of the advantages and disadvantages associated with non-specific and specific detectors may assist the analyst in the decision-making process.

Non-Specific Multidetector Analytical System

Advantages

- Somewhat lower equipment cost than GC-MS
- Less sample volume required for analysis
- More sensitive
  - ECD may be 1000 times more sensitive than GC-MS

Disadvantages

- Multiple detectors to calibrate
- Compound identification not positive
- Lengthy data interpretation (one hour each for analysis and data reduction)
- Interference(s) from co-eluting compound(s)
- Cannot identify unknown compounds
  - outside range of calibration
  - without standards
- Does not differentiate targeted compounds from interfering compounds

Specific Detector Analytical System

GC-MS-SIM

Advantages	Disadvantages
o positive compound identification	o can't identify non-specified compounds (ions)
o greater sensitivity than GC-MS-SCAN	o somewhat greater equipment cost than multidetector GC
o less operator interpretation than for multidetector GC	o greater sample volume required than for multidetector GC
o can resolve co-eluting peaks	o universality of detector sacrificed to achieve enhancement in sensitivity
o more specific than the multidetector GC	

GC-MS-SCAN

o positive compound identification	o lower sensitivity than GC-MS-SIM
o can identify all compounds	o greater sample volume required than for multidetector GC
o less operator interpretation	o somewhat greater equipment cost than multidetector GC
o can resolve co-eluting peaks	

The analytical finish for the measurement chosen by the analyst should provide a definitive identification and a precise quantitation of volatile organics. In a large part, the actual approach to these two objectives is subject to equipment availability. Figure 1 indicates some of the favorite options that are used as an analytical finish. The GC-MS-SCAN option uses a capillary column GC coupled to a MS operated in a scanning mode and supported by spectral library search routines. This option offers the nearest approximation to unambiguous identification and covers a wide range of compounds as defined by the completeness of the spectral library. GC-MS-SIM mode is limited to a set of target compounds which are user defined and is more sensitive than GC-MS-SCAN by virtue of the longer dwell times at the restricted number of m/z values. Both these techniques, but especially the GC-MS-SIM option, can use a supplemental general non-specific detector to verify/identify the presence of VOCs. Finally, the option labelled GC-multidetector system uses a

combination of retention time and multiple general detector verification to identify compounds. However, interference due to nearly identical retention times can affect system quantitation when using this option.

Due to the low concentrations of VOCs encountered in urban air (typically less than 4 ppbv and the majority below 1 ppbv) along with their complicated chromatograms, Method TO-14 strongly recommends the specific detectors (GC-MS-SCAN-SIM) for positive identification and for primary quantitation to ensure that high-quality ambient data is acquired.

For the experienced analyst whose analytical system is limited to the non-specific detectors, Section 10.3 does provide guidelines and example chromatograms showing typical retention times and calibration response factors, and utilizing the non-specific detectors (GC-FID-ECD-PID) analytical system as the primary quantitative technique.

4. Significance

4.1 VOCs enter the atmosphere from a variety of sources, including petroleum refineries, synthetic organic chemical plants, natural gas processing plants, and automobile exhaust. Many of these VOCs are acutely toxic; therefore, their determination in ambient air is necessary to assess human health impacts.

4.2 Conventional methods for VOC determination use solid sorbent sampling techniques. The most widely used solid sorbent is Tenax®. An air sample is drawn through a Tenax®-filled cartridge where certain VOCs are trapped on the polymer. The sample cartridge is transferred to a laboratory and analyzed by GC-MS.

4.3 VOCs can also be successfully collected in stainless steel canisters. Collection of ambient air samples in canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples; (4) unattended sample collection; (5) analysis of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of

samples by several analytical systems. However, care must be exercised in selecting, cleaning, and handling sample canisters and sampling apparatus to avoid losses or contamination of the samples. Contamination is a critical issue with canister-based sampling because the canister is the last element in the sampling train.

4.4 Interior surfaces of the canisters are treated by the SUMMA® passivation process, in which a pure chrome-nickel oxide is formed on the surface. This type of vessel has been used in the past for sample collection and has demonstrated sample storage stability of many specific organic compounds.

4.5 This method can be applied to sampling and analysis of not only VOCs, but also some selected semivolatile organic compounds (SVOCs). The term "semivolatile organic compounds" is used to broadly describe organic compounds that are too volatile to be collected by filtration air sampling but not volatile enough for thermal desorption from solid sorbents. SVOCs can generally be classified as those with saturation vapor pressures at 25°C between 10^{-1} and 10^{-7} mm Hg. VOCs are generally classified as those organics having saturated vapor pressures at 25°C greater than 10^{-1} mm Hg.

5. Definitions

Note: Definitions used in this document and in any user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Methods D1356, E260, and E355. All abbreviations and symbols within this method are defined at point of use.

5.1 Absolute canister pressure = Pg+Pa, where Pg = gauge pressure in the canister (kPa, psi) and Pa = barometric pressure (see 5.2).

5.2 Absolute pressure - Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPa, mm Hg or psia.

5.3 Cryogen - A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid oxygen (bp -183.0°C) or liquid argon (bp -185.7°C).

5.4 Dynamic calibration - Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

5.5 Gauge pressure - Pressure measured above ambient atmospheric pressure (as opposed to absolute pressure). Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.

5.6 MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.

5.7 MS-SIM - The GC is coupled to a MS programmed to acquire data for only specified ions and to disregard all others. This is performed using SIM coupled to retention time discriminators. The GC-SIM analysis provides quantitative results for selected constituents of the sample gas as programmed by the user.

5.8 Megabore® column - Chromatographic column having an internal diameter (I.D.) greater than 0.50 mm. The Megabore® column is a trademark of the J&W Scientific Co. For purposes of this method, Megabore® refers to chromatographic columns with 0.53 mm I.D.

5.9 Pressurized sampling - Collection of an air sample in a canister with a (final) canister pressure above atmospheric pressure, using a sample pump.

5.10 Qualitative accuracy - The ability of an analytical system to correctly identify compounds.

5.11 Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

5.12 Static calibration - Calibration of an analytical system using standards in a form different than the samples to be analyzed. An example of a static calibration would be injecting a small volume of a high concentration standard directly onto a GC column, bypassing the sample extraction and preconcentration portion of the analytical system.

AR303763

5.13 Subatmospheric sampling - Collection of an air sample in an evacuated canister at a (final) canister pressure below atmospheric pressure, without the assistance of a sampling pump. The canister is filled as the internal canister pressure increases to ambient or near ambient pressure. An auxiliary vacuum pump may be used as part of the sampling system to flush the inlet tubing prior to or during sample collection.

6. Interferences and Limitations

6.1 Interferences can occur in sample analysis if moisture accumulates in the dryer (see Section 10.1.1.2). An automated cleanup procedure that periodically heats the dryer to about 100°C while purging with zero air eliminates any moisture buildup. This procedure does not degrade sample integrity.

6.2 Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) should be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples. Instructions for cleaning the canisters and certifying the field sampling system are described in Sections 12.1 and 12.2, respectively.

6.3 Because the GC-MS analytical system employs a Nafion® permeable membrane dryer to remove water vapor selectively from the sample stream, polar organic compounds may permeate concurrent with the moisture molecule. Consequently, the analyst should quantitate his or her system with the specific organic constituents under examination.

7. Apparatus

7.1 Sample Collection

[Note: Subatmospheric pressure and pressurized canister sampling systems are commercially available and have been used as part of U.S. Environmental Protection Agency's Toxics Air Monitoring Stations (TAMS), Urban Air Toxic Pollutant Program (UATP), and the non-methane organic compound (NMOC) sampling and analysis program.]

7.1.1 Subatmospheric Pressure (See Figure 2 Without Metal Bellows Type Pump)

7.1.1.1 Sampling inlet line - stainless steel tubing to connect the sampler to the sample inlet.

7.1.1.2 Sample canister - leak-free stainless steel pressure vessels of desired volume (e.g., 6 L), with valve and SUMMA® passivated interior surfaces (Scientific Instrumentation Specialists, Inc., P.O. Box 8941, Moscow, ID 83843, or Anderson Samplers, Inc., 4215-C Wendell Dr., Atlanta, GA, 30336, or equivalent).

7.1.1.3 Stainless steel vacuum/pressure gauge - capable of measuring vacuum (-100 to 0 kPa or 0 to 30 in Hg) and pressure (0-206 kPa or 0-30 psig) in the sampling system (Matheson, P.O. Box 136, Morrow, GA 30200, Model 63-3704, or equivalent). Gauges should be tested clean and leak tight.

7.1.1.4 Electronic mass flow controller - capable of maintaining a constant flow rate (± 10%) over a sampling period of up to 24 hours and under conditions of changing temperature (20-40°C) and humidity (Tylan Corp., 19220 S. Normandie Ave., Torrance, CA 90502, Model FC-260, or equivalent).

7.1.1.5 Particulate matter filter - 2-um sintered stainless steel in-line filter (Nupro Co., 4800 E. 345th St., Willoughby, OH 44094, Model SS-2F-K4-2, or equivalent).

7.1.1.6 Electronic timer - for unattended sample collection (Paragon Elect. Co., 606 Parkway Blvd., P.O. Box 28, Twin Rivers, WI 54201, Model 7008-00, or equivalent).

7.1.1.7 Solenoid valve - electrically-operated, bi-stable solenoid valve (Skinner Magnelatch Valve, New Britain, CT, Model V5RAM49710, or equivalent) with Viton® seat and o-rings.

7.1.1.8 Chromatographic grade stainless steel tubing and fittings - for interconnections (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #8125,

or equivalent). All such materials in contact with sample, analyte, and support gases prior to analysis should be chromatographic grade stainless steel.

7.1.1.9 Thermostatically controlled heater - to maintain temperature inside insulated sampler enclosure above ambient temperature (Watlow Co., Pfafftown, NC, Part 04010080, or equivalent).

7.1.1.10 Heater thermostat - automatically regulates heater temperature (Elmwood Sensors, Inc., 500 Narragansett Park Dr., Pawtucket RI 02861, Model 3455-RC-0100-0222, or equivalent).

7.1.1.11 Fan - for cooling sampling system (EG&G Rotron, Woodstock, NY, Model SUZAI, or equivalent).

7.1.1.12 Fan thermostat - automatically regulates fan operation (Elmwood Sensors, Inc., Pawtucket, RI, Model 3455-RC-0100-0244, or equivalent).

7.1.1.13 Maximum-minimum thermometer - records highest and lowest temperatures during sampling period (Thomas Scientific, Brooklyn Thermometer Co., Inc., P/N 9327H30, or equivalent).

7.1.1.14 Nupro stainless steel shut-off valve - leak free, for vacuum/pressure gauge.

7.1.1.15 Auxiliary vacuum pump - continuously draws ambient air to be sampled through the inlet manifold at 10 L/min. or higher flow rate. Sample is extracted from the manifold at a lower rate, and excess air is exhausted. [Note: The use of higher inlet flow rates dilutes any contamination present in the inlet and reduces the possibility of sample contamination as a result of contact with active adsorption sites on inlet walls.]

7.1.1.16 Elapsed time meter - measures duration of sampling (Conrac, Cramer Div., Old Saybrook, CT, Type 6364, P/N 10082, or equivalent).

7.1.1.17 Optional fixed orifice, capillary, or adjustable micrometering valve - may be used in lieu of the electronic flow controller for grab samples or short duration time-integrated samples. Usually appropriate only in situations where screening samples are taken to assess future sampling activity.

7.1.2 Pressurized (Figure 2 With Metal Bellows Type Pump and Figure 3)

7.1.2.1 Sample pump - stainless steel, metal bellows type (Metal Bellows Corp., 1075 Providence Highway, Sharon, MA 02067, Model MB-151, or equivalent), capable of 2 atmospheres output pressure. Pump must be free of leaks, clean, and uncontaminated by oil or organic compounds. [Note: An alternative sampling system has been developed by Dr. R. Rasmussen, The Oregon Graduate Center (18,19) and is illustrated in Figure 3. This flow system uses, in order, a pump, a mechanical flow regulator, and a mechanical compensating flow restrictive device. In this configuration the pump is purged with a large sample flow, thereby eliminating the need for an auxiliary vacuum pump to flush the sample inlet. Interferences using this configuration have been minimal.]

7.1.2.2 Other supporting materials - all other components of the pressurized sampling system (Figure 2 with metal bellows type pump and Figure 3) are similar to components discussed in Sections 7.1.1.1 through 7.1.1.16.

7.2 Sample Analysis

7.2.1 GC-MS-SCAN Analytical System (See Figure 4)

7.2.1.1 The GC-MS-SCAN analytical system must be capable of acquiring and processing data in the MS-SCAN mode.

7.2.1.2 Gas chromatograph - capable of sub-ambient temperature programming for the oven, with other generally standard features such as gas flow regulators, automatic control of valves and integrator, etc. Flame ionization detector optional. (Hewlett Packard, Rt. 41, Avondale, PA 19311, Model 5880A, with oven temperature control and Level 4 BASIC programming, or equivalent.)

7.2.1.3 Chromatographic detector - mass-selective detector (Hewlett Packard, 3000-T Hanover St., 9B, Palo Alto, CA 94304, Model HP-5970 MS, or equivalent), equipped with computer and appropriate software (Hewlett Packard, 3000-T Hanover St., Palo Alto, CA 94304,

AR303767

HP-216 Computer, Quicksilver MS software, Pascal 3.0, mass storage 9133 HP Winchester with 3.5 inch floppy disk, or equivalent). The GC-MS is set in the SCAN mode, where the MS screens the sample for identification and quantitation of VOC species.

7.2.1.4 Cryogenic trap with temperature control assembly - refer to Section 10.1.1.3 for complete description of trap and temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, NC, 27704, Model 320-01, or equivalent).

7.2.1.5 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm^3/min, or equivalent).

7.2.1.6 Vacuum pump - general purpose laboratory pump, capable of drawing the desired sample volume through the cryogenic trap (Thomas Industries, Inc., Sheboygan, WI, Model 107BA20, or equivalent).

7.2.1.7 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Section 7.1.1.8 for description.

7.2.1.8 Chromatographic column - to provide compound separation such as shown in Table 5 (Hewlett Packard, Rt. 41, Avondale, PA 19311, OV-1 capillary column, 0.32 mm x 50 m with 0.88 um crosslinked methyl silicone coating, or equivalent).

7.2.1.9 Stainless steel vacuum/pressure gauge (optional) - capable of measuring vacuum (-101.3 to 0 kPa) and pressure (0-206 kPa) in the sampling system (Matheson, P.O. Box 136, Morrow, GA 30200, Model 63-3704, or equivalent). Gauges should be tested clean and leak tight.

7.2.1.10 Stainless steel cylinder pressure regulators - standard, two-stage cylinder regulators with pressure gauges for helium, zero air and hydrogen gas cylinders.

7.2.1.11 Gas purifiers (3) - used to remove organic impurities and moisture from gas streams (Hewlett Packard, Rt. 41, Avondale, PA, 19311, P/N [illegible], or equivalent).

AR303768

7.2.1.12 Low dead-volume tee (optional) - used to split the exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839, or equivalent).

7.2.1.13 Nafion® dryer - consisting of Nafion tubing coaxially mounted within larger tubing (Perma Pure Products, 8 Executive Drive, Toms River, NJ, 08753, Model MD-125-48, or equivalent). Refer to Section 10.1.1.2 for description.

7.2.1.14 Six-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII, or equivalent).

7.2.1.15 Chart recorder (optional) - compatible with the detector output signals to record optional FID detector response to the sample.

7.2.1.16 Electronic integrator (optional) - compatible with the detector output signal of the FID and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift.

7.2.2 GC-MS-SIM Analytical System (See Figure 4)

7.2.2.1 The GC-MS-SIM analytical system must be capable of acquiring and processing data in the MS-SIM mode.

7.2.2.2 All components of the GC-MS-SIM system are identical to Sections 7.2.1.2 through 7.2.1.16.

7.2.3 GC-Multidetector Analytical System (See Figure 5 and Figure 6)

7.2.3.1 Gas chromatograph with flame ionization and electron capture detectors (photoionization detector optional) - capable of sub-ambient temperature programming for the oven and simultaneous operation of all detectors, and with other generally standard features such as gas flow regulators, automatic control of valves and integrator, etc. (Hewlett Packard, Rt. 41, Avondale, PA 19311, Model 5880A, with oven temperature control and Level 4 BASIC programming, or equivalent).

7.2.3.2 Chart recorders - compatible with the detector output signals to record detector response to the sample.

7.2.3.3 Electronic integrator - compatible with the detector output signals and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift.

7.2.3.4 Six-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII, or equivalent).

7.2.3.5 Cryogenic trap with temperature control assembly - refer to Section 10.1.1.3 for complete description of trap and temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, NC 27704, Model 320-01, or equivalent).

7.2.3.6 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas, nitrogen make-up gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm^3/min, or equivalent).

7.2.3.7 Vacuum pump - general purpose laboratory pump, capable of drawing the desired sample volume through the cryogenic trap (see 7.2.1.6 for source and description).

7.2.3.8 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Section 7.1.1.8 for description.

7.2.3.9 Chromatographic column - to provide compound separation such as shown in Table 7. (Hewlett Packard, Rt. 41, Avondale, PA 19311, OV-1 capillary column, 0.32 mm x 50 m with 0.88 um crosslinked methyl silicone coating, or equivalent). [Note: Other columns (e.g., DB-624) can be used as long as the system meets user needs. The wider Megabore® column (i.e., 0.53 mm I.D.) is less susceptible to plugging as a result of trapped water, thus eliminating the need for a Nafion® dryer in the analytical system. The Megabore® column has sample capacity approaching that of a packed column, while retaining much of the peak resolution traits of narrower columns (i.e., 0.32 mm I.D.).

7.2.3.10 Vacuum/pressure gauges (3) - refer to Section 7.2.1.9 for description.

7.2.3.11 Cylinder pressure stainless steel regulators - standard, two-stage cylinder regulators with pressure gauges for helium, zero air, nitrogen, and hydrogen gas cylinders.

7.2.3.12 Gas purifiers (4) - used to remove organic impurities and moisture from gas streams (Hewlett-Packard, Rt. 41, Avondale, PA, 19311, P/N 19362 - 60500, or equivalent).

7.2.3.13 Low dead-volume tee - used to split (50/50) the exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839, or equivalent).

7.3 Canister Cleaning System (See Figure 7)

7.3.1 Vacuum pump - capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg.

7.3.2 Manifold - stainless steel manifold with connections for simultaneously cleaning several canisters.

7.3.3 Shut-off valve(s) - seven (7) on-off toggle valves.

7.3.4 Stainless steel vacuum gauge - capable of measuring vacuum in the manifold to an absolute pressure of 0.05 mm Hg or less.

7.3.5 Cryogenic trap (2 required) - stainless steel U-shaped open tubular trap cooled with liquid oxygen or argon to prevent contamination from back diffusion of oil from vacuum pump and to provide clean, zero air to sample canister(s).

7.3.6 Stainless steel pressure gauges (2) - 0-345 kPa (0-50 psig) to monitor zero air pressure.

7.3.7 Stainless steel flow control valve - to regulate flow of zero air into canister(s).

7.3.8 Humidifier - pressurizable water bubbler containing high performance liquid chromatography (HPLC) grade deionized water or other system capable of providing moisture to the zero air supply.

7.3.9 Isothermal oven (optional) for heating canisters (Fisher

7.4 Calibration System and Manifold (See Figure 8)

7.4.1 Calibration manifold - glass manifold, (1.25 cm I.D. x 66 cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing.

7.4.2 Humidifier - 500-mL impinger flask containing HPLC grade deionized water.

7.4.3 Electronic mass flow controllers - one 0 to 5 L/min and one 0 to 50 cm^3/min (Tylan Corporation, 23301-TS Wilmington Ave., Carson, CA, 90745, Model 2160, or equivalent).

7.4.4 Teflon® filter(s) - 47-mm Teflon® filter for particulate control, best source.

8. Reagents and Materials

8.1 Gas cylinders of helium, hydrogen, nitrogen, and zero air - ultrahigh purity grade, best source.

8.2 Gas calibration standards - cylinder(s) containing approximately 10 ppmv of each of the following compounds of interest:

vinyl chloride
vinylidene chloride
1,1,2-trichloro-1,2,2-trifluoroethane
chloroform
1,2-dichloroethane
benzene
toluene
Freon 12
methyl chloride
1,2-dichloro-1,1,2,2-tetrafluoroethane
methyl bromide
ethyl chloride
Freon 11
dichloromethane
1,1-dichloroethane
cis-1,2-dichloroethylene
1,2-dichloropropane
1,1,2-trichloroethane
1,2-dibromoethane
tetrachloroethylene
chlorobenzene
benzyl chloride
hexachloro-1,3-butadiene
methyl chloroform
carbon tetrachloride
trichloroethylene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
o-xylene
m-xylene
p-xylene
styrene
1,1,2,2-tetrachloroethane
1,3,5-trimethylbenzene
1,2,4-trimethylbenzene
m-dichlorobenzene
o-dichlorobenzene
p-dichlorobenzene
1,2,4-trichlorobenzene

The cylinder(s) should be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM). The components may be purchased in one cylinder or may be separated into different cylinders. Refer to manufacturer's specification for guidance on purchasing and mixing VOCs in gas cylinders. Those compounds purchased should match one's own target list.

8.3 Cryogen - liquid oxygen (bp -183.0°C), or liquid argon (bp -185.7°C), best source.

8.4 Gas purifiers - connected in-line between hydrogen, nitrogen, and zero air gas cylinders and system inlet line, to remove moisture and organic impurities from gas streams (Alltech Associates, 2051 Waukegan Road, Deerfield, IL, 60015, or equivalent).

8.5 Deionized water - high performance liquid chromatography (HPLC) grade, ultrahigh purity (for humidifier), best source.

8.6 4-bromofluorobenzene - used for tuning GC-MS, best source.

8.7 Hexane - for cleaning sampling system components, reagent grade, best source.

8.8 Methanol - for cleaning sampling system components, reagent grade, best source.

9. Sampling System

9.1 System Description

9.1.1 Subatmospheric Pressure Sampling [See Figure 2 (Without Metal Bellows Type Pump)]

9.1.1.1 In preparation for subatmospheric sample collection in a canister, the canister is evacuated to 0.05 mm Hg. When opened to the atmosphere containing the VOCs to be sampled, the differential pressure causes the sample to flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-integrated samples (duration of 12 to 24 hours) taken through a flow-restrictive inlet (e.g., mass flow controller, critical orifice).

9.1.1.2 With a critical orifice flow restrictor, there will be a decrease in the flow rate as the pressure approaches atmospheric. However, with a mass flow controller, the subatmospheric sampling system can maintain a constant flow rate from full vacuum to within about 7 kPa (1.0 psi) or less below ambient pressure.

9.1.2 Pressurized Sampling [See Figure 2 (With Metal Bellows Type Pump)]

9.1.2.1 Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control arrangement to achieve a typical 103-206 kPa (15-30 psig) final canister pressure. For example; a 6-liter evacuated canister can be filled at 10 cm^3/min for 24 hours to achieve a final pressure of about 144 kPa (21 psig).

9.1.2.2 In pressurized canister sampling, a metal bellows type pump draws in ambient air from the sampling manifold to fill and pressurize the sample canister.

9.1.3 All Samplers

9.1.3.1 A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled (to about 88.1 kPa for subatmospheric pressure sampling or to about one atmosphere above ambient pressure for pressurized sampling) over the desired sample period. The flow rate can be calculated by

$$F = \frac{P \times V}{T \times 60}$$

where:

F = flow rate (cm^3/min).

P = final canister pressure, atmospheres absolute. P is approximately equal to

$$\frac{\text{kPa gauge}}{101.2} + 1$$

V = volume of the canister (cm^3).

T = sample period (hours).

For example, if a 6-L canister is to be filled to 202 kPa (2 atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by

$$F = \frac{2 \times 6000}{24 \times 60} = 8.3 \text{ cm}^3/\text{min}$$

9.1.3.2 For automatic operation, the timer is wired to start and stop the pump at appropriate times for the desired sample period. The timer must also control the solenoid valve, to open the valve when starting the pump and close the valve when stopping the pump.

9.1.3.3 The use of the Skinner Magnelatch valve avoids any substantial temperature rise that would occur with a conventional, normally closed solenoid valve that would have to be energized during the entire sample period. The temperature rise in the valve could cause outgassing of organic compounds from the Viton valve seat material. The Skinner Magnelatch valve requires only a brief electrical pulse to open or close at the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either with an electronic timer that can be programmed for short (5 to 60 seconds) ON periods, or with a conventional mechanical timer and a special pulse circuit. A simple electrical pulse circuit for operating the Skinner Magnelatch solenoid valve with a conventional mechanical timer is illustrated in Figure 9(a). However, with this simple circuit, the valve may operate unreliably during brief power interruptions or if the timer is manually switched on and off too fast. A better circuit incorporating a time-delay relay to provide more reliable valve operation is shown in Figure 9(b).

9.1.3.4 The connecting lines between the sample inlet and the canister should be as short as possible to minimize their volume. The flow rate into the canister should remain relatively constant over the entire sampling period. If a critical orifice is used, some drop in the flow rate may occur near the end of the sample period as the canister pressure approaches the final calculated pressure.

9.1.3.5 As an option, a second electronic timer (see Section 7.1.1.6) may be used to start the auxiliary pump several hours prior to the sampling period to flush and condition the inlet line.

9.1.3.6 Prior to field use, each sampling system must pass a humid zero air certification (see Section 12.2.2). All plumbing should be checked carefully for leaks. The canisters must also pass a humid zero air certification before use (see Section 12.1).

9.2 Sampling Procedure

9.2.1 The sample canister should be cleaned and tested according to the procedure in Section 12.1.

9.2.2 A sample collection system is assembled as shown in Figure 2 (and Figure 3) and must meet certification requirements as outlined in Section 12.2.3. [Note: The sampling system should be contained in an appropriate enclosure.]

9.2.3 Prior to locating the sampling system, the user may want to perform "screening analyses" using a portable GC system, as outlined in Appendix B, to determine potential volatile organics present and potential "hot spots." The information gathered from the portable GC screening analysis would be used in developing a monitoring protocol, which includes the sampling system location, based upon the "screening analysis" results.

9.2.4 After "screening analysis," the sampling system is located. Temperatures of ambient air and sampler box interior are recorded on canister sampling field data sheet (Figure 10). [Note: The following discussion is related to Figure 2.]

9.2.5 To verify correct sample flow, a "practice" (evacuated) canister is used in the sampling system. [Note: For a subatmospheric sampler, the flow meter and practice canister are needed. For the pump-driven system, the practice canister is not needed, as the flow can be measured at the outlet of the system.] A certified mass flow meter is attached to the inlet line of the manifold, just in front of the filter. The canister is opened. The sampler is turned on and the reading of the certified mass flow meter is compared to the sampler mass flow controller. The values should agree within ±10%. If not, the sampler mass flow meter needs to be recalibrated or there is a leak in the system. This should be investigated and corrected. [Note: Mass flow meter readings may drift. Check the zero reading carefully and add or subtract the zero reading when reading or adjusting the sampler flow rate, to compensate for any zero drift.] After two minutes, the desired canister flow rate is adjusted to the proper value (as indicated by the certified mass flow meter) by the sampler flow control unit controller (e.g., 3.5 cm^3/min for 24 hr, 7.0 cm^3/min for 12 hr). Record final flow under "CANISTER FLOW RATE," Figure 10.

9.2.6 The sampler is turned off and the elapsed time meter is reset to 000.0. Note: Any time the sampler is turned off, wait at least 30 seconds to turn the sampler back on.

9.2.7 The "practice" canister and certified mass flow meter are disconnected and a clean certified (see Section 12.1) canister is attached to the system.

9.2.8 The canister valve and vacuum/pressure gauge valve are opened.

9.2.9 Pressure/vacuum in the canister is recorded on the canister sampling field data sheet (Figure 10) as indicated by the sampler vacuum/pressure gauge.

9.2.10 The vacuum/pressure gauge valve is closed and the maximum-minimum thermometer is reset to current temperature. Time of day and elapsed time meter readings are recorded on the canister sampling field data sheet.

9.2.11 The electronic timer is set to begin and stop the sampling period at the appropriate times. Sampling commences and stops by the programmed electronic timer.

9.2.12 After the desired sampling period, the maximum, minimum, current interior temperature and current ambient temperature are recorded on the sampling field data sheet. The current reading from the flow controller is recorded.

9.2.13 At the end of the sampling period, the vacuum/pressure gauge valve on the sampler is briefly opened and closed and the pressure/vacuum is recorded on the sampling field data sheet. Pressure should be close to desired pressure. [Note: For a subatmospheric sampling system, if the canister is at atmospheric pressure when the field final pressure check is performed, the sampling period may be suspect. This information should be noted on the sampling field data sheet.] Time of day and elapsed time meter readings are also recorded.

9.2.14 The canister valve is closed. The sampling line is disconnected from the canister and the canister is removed from the system. For a subatmospheric system, a certified mass flow meter is once again connected to the inlet manifold in front of the in-line filter and a "practice" canister is attached to the Magnelatch valve of the sampling system. The final flow rate is recorded on the canister sampling field data sheet (see Figure 10). [Note: For a pressurized system, the final flow may be measured directly.] The sampler is turned off.

9.2.15 An identification tag is attached to the canister. Canister serial number, sample number, location, and date are recorded on the tag.

10. Analytical System (See Figures 4, 5 and 6)

10.1 System Description

10.1.1 GC-MS-SCAN System

10.1.1.1 The analytical system is comprised of a GC equipped with a mass-selective detector set in the SCAN mode (see Figure 4). All ions are scanned by the MS repeatedly during the

GC run. The system includes a computer and appropriate software for data acquisition, data reduction, and data reporting. A 400 cm^3 air sample is collected from the canister into the analytical system. The sample air is first passed through a Nafion® dryer, through the 6-port chromatographic valve, then routed into a cryogenic trap. [Note: While the GC-multidetector analytical system does not employ a Nafion® dryer for drying the sample gas stream, it is used here because the GC-MS system utilizes a larger sample volume and is far more sensitive to excessive moisture than the GC-multidetector analytical system. Moisture can adversely affect detector precision. The Nafion® dryer also prevents freezing of moisture on the 0.32 mm I.D. column, which may cause column blockage and possible breakage.] The trap is heated (-160°C to 120°C in 60 sec) and the analyte is injected onto the OV-1 capillary column (0.32 mm x 50 m). [Note: Rapid heating of the trap provides efficient transfer of the sample components onto the gas chromatographic column.] Upon sample injection onto the column, the MS computer is signaled by the GC computer to begin detection of compounds which elute from the column. The gas stream from the GC is scanned within a preselected range of atomic mass units (amu). For detection of compounds in Table 1, the range should be 18 to 250 amu, resulting in a 1.5 Hz repetition rate. Six (6) scans per eluting chromatographic peak are provided at this rate. The 10-15 largest peaks are chosen by an automated data reduction program, the three scans nearest the peak apex are averaged, and a background subtraction is performed. A library search is then performed and the top ten best matches for each peak are listed. A qualitative characterization

of the sample is provided by this procedure. A typical chromatogram of VOCs determined by GC-MS-SCAN is illustrated in Figure 11(a).

10.1.1.2 A Nafion® permeable membrane dryer is used to remove water vapor selectively from the sample stream. The permeable membrane consists of Nafion® tubing (a copolymer of tetrafluoroethylene and fluorosulfonyl monomer) that is coaxially mounted within larger tubing. The sample stream is passed through the interior of the Nafion® tubing, allowing water (and other light, polar compounds) to permeate through the walls into a dry air purge stream flowing through the annular space between the Nafion® and outer tubing. [Note: To prevent excessive moisture build-up and any memory effects in the dryer, a clean-up procedure involving periodic heating of the dryer (100°C for 20 minutes) while purging with dry zero air (500 cm^3/min) should be implemented as part of the user's SOP manual. The clean-up procedure is repeated during each analysis (see Section 14, reference 7). Recent studies have indicated no substantial loss of targeted VOCs utilizing the above clean-up procedure (7). This cleanup procedure is particularly useful when employing cryogenic preconcentration of VOCs with subsequent GC analysis using a 0.32 mm I.D. column because excess accumulated water can cause trap and column blockage and also adversely affect detector precision. In addition, the improvement in water removal from the sampling stream will allow analyses of much larger volumes of sample air in the event that greater system sensitivity is required for targeted compounds.]

10.1.1.3 The packed metal tubing used for reduced temperature trapping of VOCs is shown in Figure 12. The cooling unit is comprised of a 0.32 cm outside diameter (O.D.) nickel tubing loop packed with 60-80 mesh Pyrex® beads (Nutech Model 320-01, or equivalent). The nickel tubing loop is wound onto a cylindrically formed tube heater (250 watt). A cartridge heater (25 watt) is sandwiched between pieces of aluminum plate at the trap inlet and outlet to provide additional heat to eliminate cold spots in the transfer tubing. During operation, the trap is inside a two-section stainless steel shell which is well insulated. Rapid heating (-150 to +100°C in 55 s) is accomplished by direct thermal contact between the heater and the trap tubing. Cooling is achieved by vaporization of the cryogen. In the shell, efficient cooling (+120 to -150°C in 225 s) is facilitated by confining the vaporized cryogen to the small open volume surrounding the trap assembly. The trap assembly and chromatographic valve are mounted on a baseplate fitted into the injection and auxiliary zones of the GC on an insulated pad directly above the column oven when used with the Hewlett-Packard 5880 GC. [Note: Alternative trap assembly and connection to the GC may be used depending upon user's requirements.] The carrier gas line is connected to the injection end of the analytical column with a zero-dead-volume fitting that is usually held in the heated zone above the GC oven. A 15 cm x 15 cm x 24 cm aluminum box is fitted over the sample handling elements to complete the package. Vaporized cryogen is vented through the top of the box.

10.1.1.4 As an option, the analyst may wish to split the gas stream exiting the column with a low dead-volume tee, passing one-third of the sample gas (1.0 mL/min) to the mass-selective detector and the remaining two-thirds (2.0 mL/min) through a flame ionization detector, as illustrated as an option in Figure 4. The use of the specific detector (MS-SCAN) coupled with the non-specific detector (FID) enables enhancement of data acquired from a single analysis. In particular, the FID provides the user:

- o Semi-real time picture of the progress of the analytical scheme;
- o Confirmation by the concurrent MS analysis of other labs that can provide only FID results; and
- o Ability to compare GC-FID with other analytical laboratories with only GC-FID capability.

10.1.2 GC-MS-SIM System

10.1.2.1 The analytical system is comprised of a GC equipped with an OV-1 capillary column (0.32 mm x 50 m) and a mass-selective detector set in the SIM mode (see Figure 4). The GC-MS is set up for automatic, repetitive analysis. The system is programmed to acquire data for only the target compounds and to disregard all others. The sensitivity is 0.1 ppbv for a 250 cm^3 air sample with analytical precision of about 5% relative standard deviation. Concentration of compounds based upon a previously installed calibration table is reported by an automated data reduction program. A Nafion® dryer is also employed by this analytical system prior to cryogenic preconcentration; therefore, many polar compounds are not identified by this procedure.

10.1.2.2 SIM analysis is based on a combination of retention times and relative abundances of selected ions (see Table 2). These qualifiers are stored on the hard disk of the GC-MS computer and are applied for identification of each chromatographic peak. The retention time qualifier is determined to be ± 0.10 minute of the library retention time of the compound. The acceptance level for relative abundance is determined to be ± 15% of the expected abundance, except for vinyl chloride and methylene chloride, which is determined to be ± 25%. Three ions are measured for most of the forty compounds. When compound identification is made by the computer, any peak that fails any of the qualifying tests is flagged (e.g., with an *). All the data should be manually examined by the analyst to determine the reason for the flag and whether the compound should be reported as found. While this adds some subjective judgment to the analysis, computer-generated identification problems can be clarified by an experienced operator. Manual inspection of the quantitative results should also be performed to verify concentrations outside the expected range. A typical chromatogram of VOCs determined by GC-MS-SIM mode is illustrated in Figure 11(b).

10.1.3 GC-Multidetector (GC-FID-ECD) System with Optional PID

10.1.3.1 The analytical system (see Figure 5) is comprised of a gas chromatograph equipped with a capillary column and electron capture and flame ionization detectors (see Figure 5). In typical operation, sample air from pressurized canisters is vented past the inlet to the analytical system from the canister at a flow rate of 75 cm^3/min. For analysis, only 35 cm^3/min of sample gas is used, while excess

is vented to the atmosphere. Sub-ambient pressure canisters are connected directly to the inlet. The sample gas stream is routed through a six port chromatographic valve and into the cryogenic trap for a total sample volume of 490 cm^3. [Note: This represents a 14 minute sampling period at a rate of 35 cm^3/min.] The trap (see Section 10.1.1.3) is cooled to -150°C by controlled release of a cryogen. VOCs and SVOCs are condensed on the trap surface while N_2, O_2, and other sample components are passed to the pump. After the organic compounds are concentrated, the valve is switched and the trap is heated. The revolatilized compounds are transported by helium carrier gas at a rate of 4 cm^3/min to the head of the Megabore® OV-1 capillary column (0.53 mm x 30 m). Since the column initial temperature is at -50°C, the VOCs and SVOCs are cryofocussed on the head of the column. Then, the oven temperature is programmed to increase and the VOCs/SVOCs in the carrier gas are chromatographically separated. The carrier gas containing the separated VOCs/SVOCs is then directed to two parallel detectors at a flow rate of 2 cm^3/min each. The detectors sense the presence of the speciated VOCs/SVOCs, and the response is recorded by either a strip chart recorder or a data processing unit.

10.1.3.2 Typical chromatograms of VOCs determined by the GC-FID-ECD analytical system are illustrated in Figures 11(c) and 11(d), respectively.

10.1.3.3 Helium is used as the carrier gas (4 cm^3/min) to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs through the Megabore® GC column. Moisture and organic impurities are removed from the helium gas stream by a chemical purifier installed in the GC (see

Section 7.2.1.11). After exiting the OV-1 Megabore® column, the carrier gas stream is split to the two detectors at rates of 2 cm^3/min each.

10.1.3.4 Gas scrubbers containing Drierite® or silica gel and 5A molecular sieve are used to remove moisture and organic impurities from the zero air, hydrogen, and nitrogen gas streams. [Note: Purity of gas purifiers is checked prior to use by passing humid zero-air through the gas purifier and analyzing according to Section 12.2.2.]

10.1.3.5 All lines should be kept as short as practical. All tubing used for the system should be chromatographic grade stainless steel connected with stainless steel fittings. After assembly, the system should be checked for leaks according to manufacturer's specifications.

10.1.3.6 The FID burner air, hydrogen, nitrogen (make-up), and helium (carrier) flow rates should be set according to the manufacturer's instructions to obtain an optimal FID response while maintaining a stable flame throughout the analysis. Typical flow rates are: burner air, 450 cm^3/min; hydrogen, 30 cm^3/min; nitrogen, 30 cm^3/min; helium, 2 cm^3/min.

10.1.3.7 The ECD nitrogen make-up gas and helium carrier flow rates should be set according to manufacturer's instructions to obtain an optimal ECD response. Typical flow rates are: nitrogen, 76 cm^3/min and helium, 2 cm^3/min.

10.1.3.8 The GC-FID-ECD could be modified to include a PID (see Figure 6) for increased sensitivity (20). In the photoionization process, a molecule is ionized by ultraviolet light as follows: R + hv --> R + e-, where R+ is the ionized species and a photon is represented by hv, with energy less than or equal to the ionization potential of

the molecule. Generally all species with an ionization potential less than the ionization energy of the lamp are detected. Because the ionization potential of all major components of air (O_2, N_2, CO, CO_2, and H_2O) is greater than the ionization energy of lamps in general use, they are not detected. The sensor is comprised of an argon-filled, ultraviolet (UV) light source where a portion of the organic vapors are ionized in the gas stream. A pair of electrodes are contained in a chamber adjacent to the sensor. When a positive potential is applied to the electrodes, any ions formed by the absorption of UV light are driven by the created electronic field to the cathode, and the current (proportional to the organic vapor concentration) is measured. The PID is generally used for compounds having ionization potentials less than the ratings of the ultraviolet lamps. This detector is used for determination of most chlorinated and oxygenated hydrocarbons, aromatic compounds, and high molecular weight aliphatic compounds. Because the PID is insensitive to methane, ethane, carbon monoxide, carbon dioxide, and water vapor, it is an excellent detector. The electron volt rating is applied specifically to the wavelength of the most intense emission line of the lamp's output spectrum. Some compounds with ionization potentials above the lamp rating can still be detected due to the presence of small quantities of more intense light. A typical system configuration associated with the GC-FID-ECD-PID is illustrated in Figure 6. This system is currently being used in EPA's FY-88 Urban Air Toxics Monitoring Program.

10.2 GC-MS-SCAN-SIM System Performance Criteria

10.2.1 GC-MS System Operation

10.2.1.1 Prior to analysis, the GC-MS system is assembled and checked according to manufacturer's instructions.

10.2.1.2 Table 3.0 outlines general operating conditions for the GC-MS-SCAN-SIM system with optional FID.

10.2.1.3 The GC-MS system is first challenged with humid zero air (see Section 11.2.2).

10.2.1.4 The GC-MS and optional FID system is acceptable if it contains less than 0.2 ppbv of targeted VOCs.

10.2.2 Daily GC-MS Tuning (See Figure 13)

10.2.2.1 At the beginning of each day or prior to a calibration, the GC-MS system must be tuned to verify that acceptable performance criteria are achieved.

10.2.2.2 For tuning the GC-MS, a cylinder containing 4-bromofluorobenzene is introduced via a sample loop valve injection system. [Note: Some systems allow auto-tuning to facilitate this process.] The key ions and ion abundance criteria that must be met are illustrated in Table 4. Analysis should not begin until all those criteria are met.

10.2.2.3 The GC-MS tuning standard could also be used to assess GC column performance (chromatographic check) and as an internal standard. Obtain a background correction mass spectra of 4-bromofluorobenzene and check that all key ions criteria are met. If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

10.2.2.4 The performance criteria must be achieved before any samples, blanks or standards are analyzed. If

any key ion abundance observed for the daily 4-bromofluorobenzene mass tuning check differs by more than 10% absolute abundance from that observed during the previous daily tuning, the instrument must be retuned or the sample and/or calibration gases reanalyzed until the above condition is met.

10.2.3 GC-MS Calibration (See Figure 13)

[Note: Initial and routine calibration procedures are illustrated in Figure 13.]

10.2.3.1 Initial Calibration - Initially, a multipoint dynamic calibration (three levels plus humid zero air) is performed on the GC-MS system, before sample analysis, with the assistance of a calibration system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs in pressurized cylinders [containing a mixture of the targeted VOCs at nominal concentrations of 10 ppmv in nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. The contents of the working standard cylinder(s) are metered (2 cm^3/min) into the heated mixing chamber where they are mixed with a 2-L/min humidified zero air gas stream to achieve a nominal 10 ppbv per compound calibration mixture (see Figure 8). This nominal 10 ppbv standard mixture is allowed to flow and equilibrate for a minimum of 30 minutes. After the equilibration period, the gas standard mixture is sampled and analyzed by the real-time GC-MS system [see Figure 8(a) and Section 7.2.1]. The results of the analyses are averaged, flow audits are performed on the mass flow meters and the calculated concentration compared to generated values. After the GC-MS is calibrated at three concentration levels, a second humid zero air sample is passed through the system and analyzed. The second humid zero air test is used to verify that the GC-MS system is certified clean (less than 0.2 ppbv of target compounds).

10.2.3.2 As an alternative, a multipoint humid static calibration (three levels plus zero humid air) can be performed on the GC-MS system. During the humid static calibration analyses, three (3) SUMMA® passivated canisters are filled each at a different concentration between 1-20 ppbv from the calibration manifold using a pump and mass flow control arrangement [see Figure 8(c)]. The canisters are then delivered to the GC-MS to serve as calibration standards. The canisters are analyzed by the MS in the SIM mode, each analyzed twice. The expected retention time and ion abundance (see Table 2 and Table 5) are used to verify proper operation of the GC-MS system. A calibration response factor is determined for each analyte, as illustrated in Table 5, and the computer calibration table is updated with this information, as illustrated in Table 6.

10.2.3.3 Routine Calibration - The GC-MS system is calibrated daily (and before sample analysis) with a one-point calibration. The GC-MS system is calibrated either with the dynamic calibration procedure [see Figure 8(a)] or with a 6-L SUMMA® passivated canister filled with humid calibration standards from the calibration manifold (see Section 10.2.3.2). After the single point calibration, the GC-MS analytical system is challenged with a humidified zero gas stream to insure the analytical system returns to specification (less than 0.2 ppbv of selective organics).

10.3 GC-FID-ECD System Performance Criteria (With Optional PID System) (See Figure 14)

10.3.1 Humid Zero Air Certification

10.3.1.1 Before system calibration and sample analysis, the GC-FID-ECD analytical system is assembled and checked according to manufacturer's instructions.

10.3.1.2 The GC-FID-ECD system is first challenged with humid zero air (see Section 12.2.2) and monitored.

10.3.1.3 Analytical systems contaminated with less than 0.2 ppbv of targeted VOCs are acceptable.

10.3.2 GC Retention Time Windows Determination (See Table 7)

10.3.2.1 Before analysis can be performed, the retention time windows must be established for each analyte.

10.3.2.2 Make sure the GC system is within optimum operating conditions.

10.3.2.3 Make three injections of the standard containing all compounds for retention time window determination. [Note: The retention time window must be established for each analyte every 72 hours during continuous operation.]

10.3.2.4 Calculate the standard deviation of the three absolute retention times for each single component standard. The retention window is defined as the mean plus or minus three times the standard deviation of the individual retention times for each standard. In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a closely-eluting, similar compound to develop a valid retention time window.

10.3.2.5 The laboratory must calculate retention time windows for each standard (see Table 7) on each GC column, whenever a new GC column is installed or when major components of the GC are changed. The data must be noted and retained in a notebook by the laboratory as part of the user SOP and as a quality assurance check of the analytical system.

10.3.3 GC Calibration

[Note: Initial and routine calibration procedures are illustrated in Figure 14.]

10.3.3.1 Initial Calibration - Initially, a multipoint dynamic calibration (three levels plus humid zero air) is performed on the GC-FID-ECD system, before sample analysis, with the assistance of a calibration system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs in pressurized cylinders [containing a mixture of the targeted VOCs at nominal concentrations of 10 ppmv in nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. The contents of the working standard cylinders are metered (2 cm^3/min) into the heated mixing chamber where they are mixed with a 2-L/min humidified zero air stream to achieve a nominal 10 ppbv per compound calibration mixture (see Figure 8). This nominal 10 ppbv standard mixture is allowed to flow and equilibrate for an appropriate amount of time. After the equilibration period, the gas standard mixture is sampled and analyzed by the GC-MS system [see Figure 8(a)]. The results of the analyses are averaged, flow audits are performed on the mass flow controllers used to generate the standards and the appropriate response factors (concentration/area counts) are calculated for each compound, as illustrated in Table 5. [Note: GC-FIDs are linear in the 1-20 ppbv range and may not require repeated multipoint calibrations; whereas, the GC-ECD will require frequent linearity evaluation.] Table 5 outlines typical calibration response factors

and retention times for 40 VOCs. After the GC-FID-ECD is calibrated at the three concentration levels, a second humid zero air sample is passed through the system and analyzed. The second humid zero air test is used to verify that the GC-FID-ECD system is certified clean (less than 0.2 ppbv of target compounds).

10.3.3.2 Routine Calibration - A one point calibration is performed daily on the analytical system to verify the initial multipoint calibration (see Section 10.3.3.1). The analyzers (GC-FID-ECD) are calibrated (before sample analysis) using the static calibration procedures (see Section 10.2.3.2) involving pressurized gas cylinders containing low concentrations of the targeted VOCs (10 ppbv) in nitrogen. After calibration, humid zero air is once again passed through the analytical system to verify residual VOCs are not present.

10.3.4 GC-FID-ECD-PID System Performance Criteria

10.3.4.1 As an option, the user may wish to include a photoionization detector (PID) to assist in peak identification and increase sensitivity.

10.3.4.2 This analytical system is presently being used in U.S. Environmental Protection Agency's Urban Air Toxic Pollutant Program (UATP).

10.3.4.3 Preparation of the GC-FID-ECD-PID analytical system is identical to the GC-FID-ECD system (see Section 10.3).

10.3.4.4 Table 8 outlines typical retention times (minutes) for selected organics using the GC-FID-ECD-PID analytical system.

10.4 Analytical Procedures

10.4.1 Canister Receipt

10.4.1.1 The overall condition of each sample canister is observed. Each canister should be received with an attached sample identification tag.

10.4.1.2 Each canister is recorded in the dedicated laboratory logbook. Also noted on the identification tag are date received and initials of recipient.

10.4.1.3 The pressure of the canister is checked by attaching a pressure gauge to the canister inlet. The canister valve is opened briefly and the pressure (kPa, psig) is recorded. [Note: If pressure is <83 kPa (<12 psig), the user may wish to pressurize the canisters, as an option, with zero grade nitrogen up to 137 kPa (20 psig) to ensure that enough sample is available for analysis. However, pressurizing the canister can introduce additional error, increase the minimum detection limit (MDL), and is time consuming. The user should weigh these limitations as part of his program objectives before pressurizing.] Final cylinder pressure is recorded on canister sampling field data sheet (see Figure 10).

10.4.1.4 If the canister pressure is increased, a dilution factor (DF) is calculated and recorded on the sampling data sheet.

$$DF = \frac{Y_a}{X_a}$$

where:

X_a = canister pressure (kPa, psia) absolute before dilution.

Y_a = canister pressure (kPa, psia) absolute after dilution.

After sample analysis, detected VOC concentrations are multiplied by the dilution factor to determine concentration in the sampled air.

10.4.2 GC-MS-SCAN Analysis (With Optional FID System)

10.4.2.1 The analytical system should be properly assembled, humid zero air certified (see Section 12.3), operated (see Table 3), and calibrated for accurate VOC determination.

10.4.2.2 The mass flow controllers are checked and adjusted to provide correct flow rates for the system.

10.4.2.3 The sample canister is connected to the inlet of the GC-MS-SCAN (with optional FID) analytical system. For pressurized samples, a mass flow controller is placed on the canister and the canister valve is opened and the canister flow is vented past a tee inlet to the analytical system at a flow of 75 cm^3/min so that 40 cm^3/min is pulled through the Nafion® dryer to the six-port chromatographic valve. [Note: Flow rate is not as important as acquiring sufficient sample volume.] Sub-ambient pressure samples are connected directly to the inlet.

10.4.2.4 The GC oven and cryogenic trap (inject position) are cooled to their set points of -50°C and -160°C, respectively.

10.4.2.5 As soon as the cryogenic trap reaches its lower set point of -160°C, the six-port chromatographic valve is turned to its fill position to initiate sample collection.

10.4.2.6 A ten minute collection period of canister sample is utilized. [Note: 40 cm^3/min x 10 min = 400 cm^3 sampled canister contents.]

10.4.2.7 After the sample is preconcentrated in the cryogenic trap, the GC sampling valve is cycled to the inject position and the cryogenic trap is heated. The trapped analytes are thermally desorbed onto the head of the OV-1 capillary column (0.31 mm I.D. x 50 m length). The GC oven is programmed to start at -50°C and after 2 min to heat to 150°C at a rate of 8°C per minute.

10.4.2.8 Upon sample injection onto the column, the MS is signaled by the computer to scan the eluting carrier gas from 18 to 250 amu, resulting in a 1.5 Hz repetition rate. This corresponds to about 6 scans per eluting chromatographic peak.

10.4.2.9 Primary identification is based upon retention time and relative abundance of eluting ions as compared to the spectral library stored on the hard disk of the GC-MS data computer.

10.4.2.10 The concentration (ppbv) is calculated using the previously established response factors (see Section 10.2.3.2), as illustrated in Table 5. [Note: If the canister is diluted before analysis, an appropriate multiplier is applied to correct for the volume dilution of the canister (Section 10.4.1.4).]

10.4.2.11 The optional FID trace allows the analyst to record the progress of the analysis.

10.4.3 GC-MS-SIM Analysis (With Optional FID System)

10.4.3.1 When the MS is placed in the SIM mode of operation, the MS monitors only preselected ions, rather than scanning all masses continuously between two mass limits.

10.4.3.2 As a result, increased sensitivity and improved quantitative analysis can be achieved.

10.4.3.3 Similar to the GC-MS-SCAN configuration, the GC-MS-SIM analysis is based on a combination of retention times and relative abundances of selected ions (see Table 2 and Table 5). These qualifiers are stored on the hard disk of the GC-MS computer and are applied for identification of each chromatographic peak. Once the GC-MS-SIM has identified the peak, a calibration response factor is used to determine the analyte's concentration.

10.4.3.4 The individual analyses are handled in three phases: data acquisition, data reduction, and data reporting. The data acquisition software is set in the SIM mode, where specific compound fragments are monitored by the MS at specific times in the analytical run. Data reduction is coordinated by the postprocessing macro program that is automatically accessed after data acquisition is completed at the end of the GC run. Resulting ion profiles are extracted, peaks are identified and integrated, and an internal integration report is generated by the program. A reconstructed ion chromatogram for hardcopy reference is prepared by the program and various parameters of interest such as time, date, and integration constants are printed. At the completion of the macro program, the data reporting software is accessed. The appropriate calibration table (see Table 9) is retrieved by the data reporting program from the computer's hard disk storage and the proper retention time and response factor parameters are applied to the macro program's integration file. With reference to certain pre-set acceptance criteria, peaks are automatically identified and quantified and a final summary report is prepared, as illustrated in Table 10.

10.4.4 GC-FID-ECD Analysis (With Optional PID System)

10.4.4.1 The analytical system should be properly assembled, humid zero air certified (see Section 12.2) and calibrated through a dynamic standard calibration procedure (see Section 10.3.2). The FID detector is lit and allowed to stabilize.

10.4.4.2 Sixty-four minutes are required for each sample analysis - 15 min for system initialization, 14 min for sample collection, 30 min for analysis, and 5 min for post-time, during which a report is printed. [Note: This may vary depending upon system configuration and programming.]

10.4.4.3 The helium and sample mass flow controllers are checked and adjusted to provide correct flow rates for the system. Helium is used to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs from the trap onto the GC column and into the FID-ECD. The hydrogen, burner air, and nitrogen flow rates should also be checked. The cryogenic trap is connected and verified to be operating properly while flowing cryogen through the system.

10.4.4.4 The sample canister is connected to the inlet of the GC-FID-ECD analytical system. The canister valve is opened and the canister flow is vented past a tee inlet to the analytical system at 75 cm^3/min using a 0-500 cm^3/min Tylan mass flow controller. During analysis, 40 cm^3/min of sample gas is pulled through the six-port chromatographic valve and routed through the trap at the appropriate time while the extra sample is vented. The VOCs are condensed in the trap while the excess flow is exhausted through an exhaust vent, which assures that the sample air flowing through the trap is at atmospheric pressure.

10.4.4.5 The six-port valve is switched to the inject position and the canister valve is closed.

10.4.4.6 The electronic integrator is started.

10.4.4.7 After the sample is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocussed on the column. Then, the oven temperature (programmed) increases and the VOCs elute from the column to the parallel FID-ECD assembly.

10.4.4.8 The peaks eluting from the detectors are identified by retention time (see Table 7 and Table 8), while peak areas are recorded in area

counts. Figures 15 and 16 illustrate typical response of the FID and ECD, respectively, for the forty (40) targeted VOCs. [Note: Refer to Table 7 for peak number and identification.]

10.4.4.9 The response factors (see Section 10.3.3.1) are multiplied by the area counts for each peak to calculate ppbv estimates for the unknown sample. If the canister is diluted before analysis, an appropriate dilution multiplier (DF) is applied to correct for the volume dilution of the canister (see Section 10.4.1.4).

10.4.4.10 Depending on the number of canisters to be analyzed, each canister is analyzed twice and the final concentrations for each analyte are the averages of the two analyses.

10.4.4.11 However, if the GC-FID-ECD analytical system discovers unexpected peaks which need further identification and attention or overlapping peaks are discovered, eliminating possible quantitation, the sample should then be subjected to a GC-MS-SCAN for positive identification and quantitation.

11. Cleaning and Certification Program

11.1 Canister Cleaning and Certification

11.1.1 All canisters must be clean and free of any contaminants before sample collection.

11.1.2 All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air. [Note: The canister cleaning system in Figure 7 can be used for this task.] The initial pressure is measured, the canister valve is closed, and the final pressure is checked after 24 hours. If leak tight, the pressure should not vary more than ± 13.8 kPa (± 2 psig) over the 24 hour period.

11.1.3 A canister cleaning system may be assembled as illustrated in Figure 7. Cryogen is added to both the vacuum pump and zero air supply traps. The manometer(s)

are connected to the manifold. The vent shut-off valve and the canister valve(s) are opened to release any remaining pressure in the canister(s). The vacuum pump is started and the vent shut-off valve is then closed and the vacuum shut-off valve is opened. The canister(s) are evacuated to < 0.05 mm Hg (for at least one hour). [Note: On a daily basis or more often if necessary, the cryogenic traps should be purged with zero air to remove any trapped water from previous canister cleaning cycles.]

11.1.4 The vacuum and vacuum/pressure gauge shut-off valves are closed and the zero air shut-off valve is opened to pressurize the canister(s) with humid zero air to approximately 206 kPa (30 psig). If a zero gas generator system is used, the flow rate may need to be limited to maintain the zero air quality.

11.1.5 The zero shut-off valve is closed and the canister(s) is allowed to vent down to atmospheric pressure through the vent shut-off valve. The vent shut-off valve is closed. Steps 11.1.3 through 11.1.5 are repeated two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.

11.1.6 At the end of the evacuation/pressurization cycle, the canister is pressurized to 206 kPa (30 psig) with humid zero air. The canister is then analyzed by a GC-MS or GC-FID-ECD analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) should not be used. As a "blank" check of the canister(s) and cleanup procedure, the final humid zero air fill of 100% of the canisters is analyzed until the cleanup system and canisters are proven reliable (less than 0.2 ppbv of targets VOCs). The check can then be reduced to a lower percentage of canisters.

11.1.7 The canister is reattached to the cleaning manifold and is then reevacuated to <0.05 mm Hg and remains in this condition until used. The canister valve is closed. The canister is removed from the cleaning system and the canister connection is capped with a stainless steel fitting.

The canister is now ready for collection of an air sample. An identification tag is attached to the neck of each canister for field notes and chain-of-custody purposes.

11.1.8 As an option to the humid zero air cleaning procedures, the canisters could be heated in an isothermal oven to 100°C during Section 11.1.3 to ensure that lower molecular weight compounds (C_2-C_8) are not retained on the walls of the canister. [Note: For sampling heavier, more complex VOC mixtures, the canisters should be heated to 250°C during Section 11.1.3.7.] Once heated, the canisters are evacuated to 0.05 mm Hg. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by the GC-FID-ECD system. Any canister that has not tested clean (less than 0.2 ppbv of targeted compounds) should not be used. Once tested clean, the canisters are reevacuated to 0.05 mm Hg and remain in the evacuated state until used.

11.2 Sampling System Cleaning and Certification

11.2.1 Cleaning Sampling System Components

11.2.1.1 Sample components are disassembled and cleaned before the sampler is assembled. Nonmetallic parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50°C. Typically, stainless steel parts and fittings are cleaned by placing them in a beaker of methanol in an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent.

11.2.1.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100°C for 12 to 24 hours.

11.2.1.3 Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.

11.2.2 Humid Zero Air Certification

[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid

zero air and humid calibration gases that pass through all active components of the sampling system. The system is "certified" if no significant additions or deletions (less than 0.2 ppbv of targeted compounds) have occurred when challenged with the test gas stream.]

11.2.2.1 The cleanliness of the sampling system is determined by testing the sampler with humid zero air without an evacuated gas cylinder, as follows.

11.2.2.2 The calibration system and manifold are assembled, as illustrated in Figure 8. The sampler (without an evacuated gas cylinder) is connected to the manifold and the zero air cylinder activated to generate a humid gas stream (2 L/min) to the calibration manifold [see Figure 8(b)].

11.2.2.3 The humid zero gas stream passes through the calibration manifold, through the sampling system (without an evacuated canister) to a GC-FID-ECD analytical system at 75 cm^3/min so that 40 cm^3/min is pulled through the six-port valve and routed through the cryogenic trap (see Section 10.2.2.1) at the appropriate time while the extra sample is vented. [Note: The exit of the sampling system (without the canister) replaces the canister in Figure 4.] After the sample (400 mL) is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocussed on the column. Then, the oven temperature (programmed) increases and the VOCs begin to elute and are detected by a GC-MS (see Section 10.2) or the GC-FID-ECD (see Section 10.3). The analytical system should not detect greater than 0.2 ppbv of targeted VOCs in order for the sampling system to pass the humid zero air certification

test. Chromatograms of a certified sampler and contaminated sampler are illustrated in Figures 17(a) and (b), respectively. If the sampler passes the humid zero air test, it is then tested with humid calibration gas standards containing selected VOCs at concentration levels expected in field sampling (e.g., 0.5 to 2 ppbv) as outlined in Section 11.2.3.

11.2.3 Sampler System Certification with Humid Calibration Gas Standards

11.2.3.1 Assemble the dynamic calibration system and manifold as illustrated in Figure 8.

11.2.3.2 Verify that the calibration system is clean (less than 0.2 ppbv of targeted compounds) by sampling a humidified gas stream, without gas calibration standards, with a previously certified clean canister (see Section 12.1).

11.2.3.3 The assembled dynamic calibration system is certified clean if less than 0.2 ppbv of targeted compounds are found.

11.2.3.4 For generating the humidified calibration standards, the calibration gas cylinder(s) (see Section 8.2) containing nominal concentrations of 10 ppmv in nitrogen of selected VOCs, are attached to the calibration system, as outlined in Section 10.2.3.1. The gas cylinders are opened and the gas mixtures are passed through 0 to 10 cm^3/min certified mass flow controllers to generate ppb levels of calibration standards.

11.2.3.5 After the appropriate equilibrium period, attach the sampling system (containing a certified evacuated canister) to the manifold, as illustrated in Figure 8(a).

11.2.3.6 Sample the dynamic calibration gas stream with the sampling system according to Section 9.2.1. [Note: To conserve generated calibration gas, bypass the canister sampling system manifold and attach the sampling system to the calibration gas stream at the inlet of the in-line filter of the sampling system so the flow will be less than 500 cm^3/min.]

11.2.3.7 Concurrent with the sampling system operation, realtime monitoring of the calibration gas stream is accomplished by the on-line GC-MS or GC-multidetector analytical system [Figure 8(b)] to provide reference concentrations of generated VOCs.

11.2.3.8 At the end of the sampling period (normally same time period used for anticipated sampling), the sampling system canister is analyzed and compared to the reference GC-MS or GC-multidetector analytical system to determine if the concentration of the targeted VOCs was increased or decreased by the sampling system.

11.2.3.9 A recovery of between 90% and 110% is expected for all targeted VOCs.

12. Performance Criteria and Quality Assurance

12.1 Standard Operating Procedures (SOPs)

12.1.1 SOPs should be generated in each laboratory describing and documenting the following activities: (1) assembly, calibration, leak check, and operation of specific sampling systems and equipment used; (2) preparation, storage, shipment, and handling of samples; (3) assembly, leak-check, calibration, and operation of the analytical system, addressing the specific equipment used; (4) canister storage and cleaning; and (5) all aspects of data recording and processing, including lists of computer hardware and software used.

12.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the laboratory personnel conducting the work.

12.2 Method Relative Accuracy and Linearity

12.2.1 Accuracy can be determined by injecting VOC standards (see Section 8.2) from an audit cylinder into a sampler. The contents are then analyzed for the components contained in the audit canister. Percent relative accuracy is calculated:

$$\% \text{ Relative Accuracy} = \frac{Y - X}{X} \times 100$$

Where: Y = Concentration of the targeted compound recovered from sampler.

X = Concentration of VOC targeted compound in the NBS-SRM or EPA-CRM audit cylinders.

12.2.2 If the relative accuracy does not fall between 90 and and 110 percent, the field sampler should be removed from use, cleaned, and recertified according to initial certification procedures outlined in Section 11.2.2 and Section 11.2.3. Historically, concentrations of carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene have sometimes been detected at lower concentrations when using parallel ECD and FID detectors. When these three compounds are present at concentrations close to calibration levels, both detectors usually agree on the reported concentrations. At concentrations below 4 ppbv, there is a problem with nonlinearity of the ECD. Plots of concentration versus peak area for calibration compounds detected by the ECD have shown that the curves are nonlinear for carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene, as illustrated in Figures 18(a) through 18(c). Other targeted ECD and FID compounds scaled linearly for the range 0 to 8 ppbv, as shown for chloroform in Figure 18(d). For compounds that are not linear over the calibration

range, area counts generally roll off between 3 and 4 ppbv. To correct for the nonlinearity of these compounds, an additional calibration step is performed. An evacuated stainless steel canister is pressurized with calibration gas at a nominal concentration of 8 ppbv. The sample is then diluted to approximately 3.5 ppbv with zero air and analyzed. The instrument response factor (ppbv/area) of the ECD for each of the three compounds is calculated for the 3.5 ppbv sample. Then, both the 3.5 ppbv and the 8 ppbv response factors are entered into the ECD calibration table. The software for the Hewlett-Packard 5880 level 4 GC is designed to accommodate multilevel calibration entries, so the correct response factors are automatically calculated for concentrations in this range.

12.3 Method Modification

12.3.1 Sampling

12.3.1.1 The sampling system for pressurized canister sampling could be modified to use a lighter, more compact pump. The pump currently being used weighs about 16 kilograms (35 lbs). Commercially available pumps that could be used as alternatives to the prescribed sampler pump are described below. Metal Bellows MB-41 pump: These pumps are cleaned at the factory; however, some precaution should be taken with the circular (4.8 cm diameter) Teflon® and stainless steel part directly under the flange. It is often dirty when received and should be cleaned before use. This part is cleaned by removing it from the pump, manually cleaning with deionized water, and placing in a vacuum oven at 100°C for at least 12 hours. Exposed parts of the pump head are also cleaned with swabs and allowed to air dry. These pumps have

proven to be very reliable; however, they are only useful up to an outlet pressure of about 137 kPa (20 psig). Neuberger Pump: Viton gaskets or seals must be specified with this pump. The "factory direct" pump is received contaminated and leaky. The pump is cleaned by disassembling the pump head (which consists of three stainless steel parts and two gaskets), cleaning the gaskets with deionized water and drying in a vacuum oven, and remachining (or manually lapping) the sealing surfaces of the stainless steel parts. The stainless steel parts are then cleaned with methanol, hexane, deionized water and heated in a vacuum oven. The cause for most of the problems with this pump has been scratches on the metal parts of the pump head. Once this rework procedure is performed, the pump is considered clean and can be used up to about 240 kPa (35 psig) output pressure. This pump is utilized in the sampling system illustrated in Figure 3.

12.3.1.2 Urban Air Toxics Sampler

The sampling system described in this method can be modified like the sampler in EPA's FY-88 Urban Air Toxics Pollutant Program. This particular sampler is described in Appendix C (see Figure 19).

12.3.2 Analysis

12.3.2.1 Inlet tubing from the calibration manifold could be heated to 50°C (same temperature as the calibration manifold) to prevent condensation on the internal walls of the system.

12.3.2.2 The analytical strategy for Method TO-14 involves positive identification and quantitation by GC-MS-SCAN-SIM mode of operation with optional FID. This is a highly specific and sensitive detection technique. Because a specific detector system (GC-MS-SCAN-SIM) is more complicated and expensive than the use of non-specific detectors

(GC-FID-ECD-PID), the analyst may want to perform a screening analysis and preliminary quantitation of VOC species in the sample, including any polar compounds, by utilizing the GC-multidetector (GC-FID-ECD-PID) analytical system prior to GC-MS analysis. This system can be used for approximate quantitation. The GC-FID-ECD-PID provides a "snapshot" of the constituents in the sample, allowing the analyst to determine:

- Extent of misidentification due to overlapping peaks,
- Whether the constituents are within the calibration range of the anticipated GC-MS-SCAN-SIM analysis or does the sample require further dilution, and
- Are there unexpected peaks which need further identification through GC-MS-SCAN or are there peaks of interest needing attention?

If unusual peaks are observed from the GC-FID-ECD-PID system, the analyst then performs a GC-MS-SCAN analysis. The GC-MS-SCAN will provide positive identification of suspect peaks from the GC-FID-ECD-PID system. If no unusual peaks are identified and only a select number of VOCs are of concern, the analyst can then proceed to GC-MS-SIM. The GC-MS-SIM is used for final quantitation of selected VOCs. Polar compounds, however, cannot be identified by the GC-MS-SIM due to the use of a Nafion® dryer to remove water from the sample prior to analysis. The dryer removes polar compounds along with the water. The analyst often has to make this decision incorporating project objectives, detection limits, equipment availability, cost and personnel capability in developing an analytical strategy. Figure 20 outlines the use of the GC-FID-ECD-PID as a "screening" approach, with the GC-MS-SCAN-SIM for final identification and quantitation.

12.4 Method Safety

This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

12.5 Quality Assurance (See Figure 21)

12.5.1 Sampling System

12.5.1.1 Section 9.2 suggests that a portable GC system be used as a "screening analysis" prior to locating fixed-site samplers (pressurized or subatmospheric).

12.5.1.2 Section 9.2 requires pre and post-sampling measurements with a certified mass flow controller for flow verification of sampling system.

12.5.1.3 Section 11.1 requires all canisters to be pressure tested to 207 kPa $\pm$ 14 kPa (30 psig $\pm$ 2 psig) over a period of 24 hours.

12.5.1.4 Section 11.1 requires that all canisters be certified clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

12.5.1.5 Section 11.2.2 requires all field sampling systems to be certified initially clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

12.5.1.6 Section 11.2.3 requires all field sampling systems to pass an initial humidified calibration gas certification [at VOC concentration levels expected in the field (e.g., 0.5 to 2 ppbv)] with a percent recovery of greater than 90.

12.5.2 GC-MS-SCAN-SIM System Performance Criteria

12.5.2.1 Section 10.2.1 requires the GC-MS analytical system to be certified clean (less than 0.2

ppbv of targeted VOCs) prior to sample analysis, through a humid zero air certification.

12.5.2.2 Section 10.2.2 requires the daily tuning of the GC-MS with 4-bromofluorobenzene (4-BFB) and that it meet the key ions and ion abundance critera (10%) outlined in Table 5.

12.5.2.3 Section 10.2.3 requires both an initial multipoint humid static calibration (three levels plus humid zero air) and a daily calibration (one point) of the GC-MS analytical system.

12.5.3 GC-Multidetector System Performance Criteria

12.5.3.1 Section 10.3.1 requires the GC-FID-ECD analytical system, prior to analysis, to be certified clean (less than 0.2 ppbv of targeted VOCs) through a humid zero air certification.

12.5.3.2 Section 10.3.2 requires that the GC-FID-ECD analytical system establish retention time windows for each analyte prior to sample analysis, when a new GC column is installed, or major components of the GC system altered since the previous determination.

12.5.3.3 Section 8.2 requires that all calibration gases be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM).

12.5.3.4 Section 10.3.2 requires that the retention time window be established throughout the course of a 72-hr analytical period.

12.5.3.5 Section 10.3.3 requires both an initial multipoint calibration (three levels plus humid zero air) and a daily calibration (one point) of the GC-FID-ECD analytical system with zero gas dilution of NBS traceable or NBS/EPA CRMs gases. [Note: Gas cylinders of VOCs at the ppm and ppb level are available for audits from the USEPA, Environmental Monitoring Systems

Laboratory, Quality Assurance Division, MD-77B, Research Triangle Park, NC 27711, (919)541-4531. Appendix A outlines five groups of audit gas cylinders available from USEPA.]

13. Acknowledgements

The determination of volatile and some semi-volatile organic compounds in ambient air is a complex task, primarily because of the wide variety of compounds of interest and the lack of standardized sampling and analytical procedures. While there are numerous procedures for sampling and analyzing VOCs/SVOCs in ambient air, this method draws upon the best aspects of each one and combines them into a standardized methodology. To that end, the following individuals contributed to the research, documentation and peer review of this manuscript.

Topic	Contact	Address	Telephone No.
Sampling System	Mr. Frank McElroy Mr. Vince Thompson	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77 Research Triangle Park, N.C. 27711	919-541-2622 919-541-3791
	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Mr. Tom Merrifield	Anderson Samplers, Inc. 4215-C Wendell Drive Atlanta, GA 30336	1-800-241-6898
	Mr. Joseph P. Krasnec	Scientific Instrumentation Specialists, Inc. P.O. Box 8941 Moscow, Idaho, 83843	208-882-3860
Analytical System			
GC-FID-ECD	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Ms. Karen D. Oliver	Northrop Services, Inc. Environmental Sciences P.O. Box 12313 Research Triangle Park, N.C. 27709	919-549-0611
GC-FID-ECD-PID	Dave-Paul Dayton JoAnn Rice	Radian Corporation P.O. Box 13000 Progress Center Research Triangle Park, N.C. 27709	919-481-0212

T014-56

AR303811

Topic	Contact	Address	Telephone No.
[redacted]-MS-SCAN-SIM	Dr. Bill McClenny Mr. Joachim Pleil	U.S. E[redacted]nmental Protection Agency Environ[redacted]tal Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3[redacted] 919-541-468[redacted]
	Mr. John V. Hawkins	Research Triangle Laboratories, Inc. P.O. Box 12507 Research Triangle Park, N.C. 27709	919-544-5775
Canister Cleaning Certification and VOC Canister Storage Stability	Mr. Vince Thompson	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77 Research Triangle Park, N.C. 27711	919-541-3791
	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Dave-Paul Dayton JoAnn Rice	Radian Corporation P.O. Box 13000 Progress Center Research Triangle Park, N.C. 27709	919-481-0212
	Dr. R.K.M. Jayanty	Research Triangle Institute P.O. Box 12194 Research Triangle Park, N.C. 27709	919-541-6000
Cryogenic Sampling Unit	Mr. Lou Ballard Mr. Pete Watson	NuTech Corporation 2806 Cheek Road Durham, N.C., 27704	919-682-0402
	Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-4680
U.S. EPA Audit Gas Standards	Mr. Bob Lampe	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77B Research Triangle Park, N.C.27711	919-541-4531

14. REFERENCES

1. K. D. Oliver, J. D. Pleil, and W. A. McClenny, "Sample Integrity of Trace Level Volatile Organic Compounds in Ambient Air Stored in SUMMA® Polished Canisters," Atmospheric Environ. 20:1403, 1986.

2. M. W. Holdren and D. L. Smith, "Stability of Volatile Organic Compounds While Stored in SUMMA® Polished Stainless Steel Canisters," Final Report, EPA Contract No. 68-02-4127, Research Triangle Park, NC, Battelle Columbus Laboratories, January, 1986.

3. Ralph M. Riggin, Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air, EPA-600/4-83-027, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1983.

4. Ralph M. Riggin, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA-600/4-84-041, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1986.

5. W. T. Winberry and N. V. Tilley, Supplement to EPA-600/4-84-041: Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA-600/4-87-006, U.S. Environmental Protection Agency, Research Triangle Park, NC, 1986.

6. W. A. McClenny, J. D Pleil, J. W. Holdren, and R. N. Smith, "Automated Cryogenic Preconcentration and Gas Chromatographic Determination of Volatile Organic Compounds," Anal. Chem. 56:2947, 1984.

7. J. D. Pleil and K. D. Oliver, "Evaluation of Various Configurations of Nafion Dryers: Water Removal from Air Samples Prior to Gas Chromatographic Analysis," EPA Contract No. 68-02-4035, Research Triangle Park, NC, Northrop Services, Inc.- Environmental Sciences, 1985.

8. K. D. Oliver and J. D. Pleil, "Automated Cryogenic Sampling and Gas Chromatographic Analysis of Ambient Vapor-Phase Organic Compounds: Procedures and Comparison Tests," EPA Contract No. 68-02-4035, Research Triangle Park, NC, Northrop Services, Inc.- Environmental Sciences, 1985.

9. W. A. McClenny and J. D. Pleil, "Automated Calibration and Analysis of VOCs with a Capillary Column Gas Chromatograph Equipped for Reduced Temperature Trapping," Proceedings of the 1984 Air Pollution Control Association Annual Meeting, San Francisco, CA, June 24-29, 1984.

10. W. A. McClenny, J. D. Pleil, T. A. Lumpkin, and K. D. Oliver, "Update on Canister-Based Samplers for VOCs," Proceedings of the 1987 EPA/APCA Symposium on Measurement of Toxic and Related Air Pollutants, May, 1987 APCA Publication VIP-8, EPA 600/9-87-010.

11. J. D. Pleil, "Automated Cryogenic Sampling and Gas Chromatographic Analysis of Ambient Vapor-Phase Organic Compounds: System Design," EPA Contract No. 68-02-2566, Research Triangle Park, NC, Northrop Services, Inc.- Environmental Sciences, 1982.

12. K. D. Oliver and J. D. Pleil, "Analysis of Canister Samples Collected During the CARB Study in August 1986," EPA Contract No. 68-02-4035, Research Triangle Park, NC, Northrop Services, Inc,- Environmental Sciences, 1987.

13. J. D. Pleil and K. D. Oliver, "Measurement of Concentration Variability of Volatile Organic Compounds in Indoor Air: Automated Operation of a Sequential Syringe Sampler and Subsequent GC/MS Analysis," EPA Contract No. 68-02-4444, Research Triangle Park, NC, Northrop Services, Inc. - Environmental Sciences, 1987.

14. J. F. Walling, "The Utility of Distributed Air Volume Sets When Sampling Ambient Air Using Solid Adsorbents," Atmospheric Environ., 18:855-859, 1984.

15. J. F. Walling, J. E. Bumgarner, J. D. Driscoll, C. M. Morris, A. E. Riley, and L. H. Wright, "Apparent Reaction Products Desorbed From Tenax Used to Sample Ambient Air," Atmospheric Environ., 20: 51-57, 1986.

16. Portable Instruments User's Manual for Monitoring VOC Sources, EPA-340/1-88-015, U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Washington, DC, June, 1986.

17. F. F. McElroy, V. L. Thompson, H. G. Richter, A Cryogenic Preconcentration - Direct FID (PDFID) Method for Measurement of NMOC in the Ambient Air, EPA-600/4-85-063, U.S. Environmental Protection Agency, Research Triangle Park, NC, August 1985.

18. R. A. Rasmussen and J. E. Lovelock, "Atmospheric Measurements Using Canister Technology," J. Geophys. Res., 83: 8369-8378, 1983.

19. R. A. Rasmussen and M.A.K. Khalil, "Atmospheric Halocarbons: Measurements and Analysis of Selected Trace Gases," Proc. NATO ASI on Atmospheric Ozone, BO: 209-231.

20. Dave-Paul Dayton and JoAnn Rice, "Development and Evaluation of a Prototype Analytical System for Measuring Air Toxics," Final Report, Radian Corporation for the U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Research Triangle Park, NC 27711, EPA Contract No. 68-02-3889, WA No. 120, November, 1987.

TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT (°C)	MELTING POINT (°C)	CAS NUMBER
Freon 12 (Dichlorodifluoromethane)	Cl_2CF_2	120.91	-29.8	-158.0	
Methyl chloride (Chloromethane)	CH_3Cl	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	$ClCF_2CClF_2$	170.93	4.1	-94.0	
Vinyl chloride (Chloroethylene)	$CH_2=CHCl$	62.50	-13.4	-1538.0	75-01-4
Methyl bromide (Bromomethane)	CH_3Br	94.94	3.6	-93.6	74-83-9
Ethyl chloride (Chloroethane)	CH_3CH_2Cl	64.52	12.3	-136.4	75-00-3
Freon 11 (Trichlorofluoromethane)	CCl_3F	137.38	23.7	-111.0	
Vinylidene chloride (1,1-Dichloroethene)	$C_2H_2Cl_2$	96.95	31.7	-122.5	75-35-4
Dichloromethane (Methylene chloride)	CH_2Cl_2	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	CF_2ClCCl_2F	187.38	47.7	-36.4	
1,1-Dichloroethane (Ethylidene chloride)	CH_3CHCl_2	98.96	57.3	-97.0	74-34-3
cis-1,2-Dichloroethylene	$CHCl=CHCl$	96.94	60.3	-80.5	
Chloroform (Trichloromethane)	$CHCl_3$	119.38	61.7	-63.5	67-66-3
1,2-Dichloroethane (Ethylene dichloride)	$ClCH_2CH_2Cl$	98.96	83.5	-35.3	107-06-2
Methyl chloroform (1,1,1-Trichloroethane)	CH_3CCl_3	133.41	74.1	-30.4	71-55-6
Benzene (Cyclohexatriene)	C_6H_6	78.12	80.1	5.5	71-43-2
Carbon tetrachloride (Tetrachloromethane)	CCl_4	153.82	76.5	-23.0	56-23-5
1,2-Dichloropropane (Propylene dichloride)	$CH_3CHClCH_2Cl$	112.99	96.4	-100.4	78-87-5
Trichloroethylene (Trichloroethene)	$ClCH=CCl_2$	131.29	87	-73.0	79-01-6
cis-1,3-Dichloropropene (cis-1,3-dichloropropylene)	$CH_3CCl=CHCl$	110.97	76		

T014-60

TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET (cont.)

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT(°C)	MELTING POINT (°C)	CAS NUMBER
trans-1,3-Dichloropropene (cis-1,3-Dichloropropylene)	$ClCH_2CH{=}CHCl$	110.97	112.0		
1,1,2-Trichloroethane (Vinyl trichloride)	$CH_2ClCHCl_2$	133.41	113.8	-36.5	79-00-5
Toluene (Methyl benzene)	$C_6H_5CH_3$	92.15	110.6	-95.0	108-88-3
1,2-Dibromoethane (Ethylene dibromide)	$BrCH_2CH_2Br$	187.88	131.3	9.8	106-93-4
Tetrachloroethylene (Perchloroethylene)	$Cl_2C{=}CCl_2$	165.83	121.1	-19.0	127-18-4
Chlorobenzene (Phenyl chloride)	C_6H_5Cl	112.56	132.0	-45.6	108-90-7
Ethylbenzene	$C_6H_5C_2H_5$	106.17	136.2	-95.0	100-41-4
m-Xylene (1,3-Dimethylbenzene)	$1,3\text{-}(CH_3)_2C_6H_4$	106.17	139.1	-47.9	
p-Xylene (1,4-Dimethylxylene)	$1,4\text{-}(CH_3)_2C_6H_4$	106.17	138.3	13.3	
Styrene (Vinyl benzene)	$C_6H_5CH{=}CH_2$	104.16	145.2	-30.6	100-42-5
1,1,2,2-Tetrachloroethane	$CHCl_2CHCl_2$	167.85	146.2	-36.0	79-34-5
o-Xylene (1,2-Dimethylbenzene)	$1,2\text{-}(CH_3)_2C_6H_4$	106.17	144.4	-25.2	
1,3,5-Trimethylbenzene (Mesitylene)	$1,3,5\text{-}(CH_3)_3C_6H_6$	120.20	164.7	-44.7	108-67-8
1,2,4-Trimethylbenzene (Pseudocumene)	$1,2,4\text{-}(CH_3)_3C_6H_6$	120.20	169.3	-43.8	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	$1,3\text{-}Cl_2C_6H_4$	147.01	173.0	-24.7	541-73-1
Benzyl chloride (α-Chlorotoluene)	$C_6H_5CH_2Cl$	126.59	179.3	-39.0	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	$1,2\text{-}Cl_2C_6H_4$	147.01	180.5	-17.0	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	$1,4\text{-}Cl_2C_6H_4$	147.01	174.0	53.1	106-46-7
1,2,4-Trichlorobenzene	$1,2,4\text{-}Cl_3C_6H_3$	181.45	213.5	17.0	120-82-1
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)					

T014-61

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Freon 12 (Dichlorodifluoromethane)	85/100 87/ 31	5.01
Methyl chloride (Chloromethane)	50/100 52/ 34	5.69
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	85/100 135/ 56 87/ 33	6.55
Vinyl chloride (Chloroethene)	62/100 27/125 64/ 32	6.71
Methyl bromide (Bromomethane)	94/100 96/ 85	7.83
Ethyl chloride (Chloroethane)	64/100 29/140 27/140	8.43
Freon 11 (Trichlorofluoromethane)	101/100 103/ 67	9.97
Vinylidene chloride (1,1-Dichloroethylene)	61/100 96/ 55 63/ 31	10.93
Dichloromethane (Methylene chloride)	49/100 84/ 65 86/ 45	11.21
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	151/100 101/140 103/ 90	11.60
1,1-Dichloroethane (Ethylidene dichloride)	63/100 27/ 64 65/ 33	12.50
cis-1,2-Dichloroethylene	61/100 96/ 60 98/ 44	13.40
Chloroform (Trichloromethane)	83/100 85/ 65 47/ 35	13.75
1,2-Dichloroethane (Ethylene dichloride)	62/100 27/ 70 64/ 31	14.39
Methyl chloroform (1,1,1-Trichloroethane)	97/100 99/ 64 61/ 61	14.62
Benzene (Cyclohexatriene)	78/100 77/ 25 50/ 35	15.04
Carbon tetrachloride (Tetrachloromethane)	117/100 119/ 97	15.18

(continued)

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont.)

Compound	Ion/Abundance (amu/% base peak)	Estimated Retention Time (min)
1,2-Dichloropropane (Propylene dichloride)	63/100 41/ 90 62/ 70	15.83
Trichloroethylene (Trichloroethene)	130/100 132/ 92 95/ 87	16.10
cis-1,3-Dichloropropene	75/100 39/ 70 77/ 30	16.96
trans-1,3-Dichloropropene (1,3 dichloro-1-propene)	75/100 39/ 70 77/ 30	17.49
1,1,2-Trichloroethane (Vinyl trichloride)	97/100 83/ 90 61/ 82	17.61
Toluene (Methyl benzene)	91/100 92/ 57	17.86
1,2-Dibromoethane (Ethylene dibromide)	107/100 109/ 96 27/115	18.48
Tetrachloroethylene (Perchloroethylene)	166/100 164/ 74 131/ 60	19.01
Chlorobenzene (Benzene chloride)	112/100 77/ 62 114/ 32	19.73
Ethylbenzene	91/100 106/ 28	20.20
m,p-Xylene(1,3/1,4-dimethylbenzene)	91/100 106/ 40	20.41
Styrene (Vinyl benzene)	104/100 78/ 60 103/ 49	20.81
1,1,2,2-Tetrachloroethane (Tetrachloroethane)	83/100 85/ 64	20.92
o-Xylene (1,2-Dimethylbenzene)	91/100 106/ 40	20.92
4-Ethyltoluene	105/100 120/ 29	22.53
1,3,5-Trimethylbenzene (Mesitylene)	105/100 120/ 42	22.65
1,2,4-Trimethylbenzene (Pseudocumene)	105/100 120/ 42	23.18
m-Dichlorobenzene (1,3-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.31

(continued)

TABLE 2. ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont.)

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Benzyl chloride (α-Chlorotoluene)	91/100 126/ 26	23.32
p-Dichlorobenzene (1,4-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.41
o-Dichlorobenzene (1,2-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.88
1,2,4-Trichlorobenzene	180/100 182/ 98 184/ 30	26.71
Hexachlorobutadiene (1,1,2,3,4,4 Hexachloro-1,3-butadiene)	225/100 227/ 66 223/ 60	27.68

TABLE 3. GENERAL GC AND MS OPERATING CONDITIONS

Chromatography	
Column	Hewlett-Packard OV-1 crosslinked methyl silicone (50 m x 0.31-mm I.D., 17 um film thickness), or equivalent
Carrier Gas	Helium (2.0 cm^3/min at 250°C)
Injection Volume	Constant (1-3 uL)
Injection Mode	Splitless
Temperature Program	
Initial Column Temperature	-50°C
Initial Hold Time	2 min
Program	8°C/min to 150°C
Final Hold Time	15 min
Mass Spectrometer	
Mass Range	18 to 250 amu
Scan Time	1 sec/scan
EI Condition	70 eV
Mass Scan	Follow manufacturer's instruction for selecting mass selective detector (MS) and selected ion monitoring (SIM) mode
Detector Mode	Multiple ion detection
FID System (Optional)	
Hydrogen Flow	30 cm^3/minute
Carrier Flow	30 cm^3/minute
Burner Air	400 cm^3/minute

TABLE 4. 4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but< 101% of mass 174
177	5 to 9% of mass 176

TABLE 5. RESPONSE FACTORS (ppbv/area count) AND EXPECTED RETENTION TIME FOR GC-MS-SIM ANALYTICAL CONFIGURATION

Compounds	Response Factor (ppbv/area count)	Expected Retention Time (minutes)
Freon 12	0.6705	5.01
Methyl chloride	4.093	5.64
Freon 114	0.4928	6.55
Vinyl chloride	2.343	6.71
Methyl bromide	2.647	7.83
Ethyl chloride	2.954	8.43
Freon 11	0.5145	9.87
Vinylidene chloride	1.037	10.93
Dichloromethane	2.255	11.21
Trichlorotrifluoroethane	0.9031	11.60
1,1-Dichloroethane	1.273	12.50
cis-1,2-Dichloroethylene	1.363	13.40
Chloroform	0.7911	13.75
1,2-Dichloroethane	1.017	14.39
Methyl chloroform	0.7078	14.62
Benzene	1.236	15.04
Carbon tetrachloride	0.5880	15.18
1,2-Dichloropropane	2.400	15.83
Trichloroethylene	1.383	16.10
cis-1,3-Dichloropropene	1.877	16.96
trans-1,3-Dichloropropene	1.338	17.49
1,1,2-Trichloroethane	1.891	17.61
Toluene	0.9406	17.86
1,2-Dibromoethane (EDB)	0.8662	18.48
Tetrachloroethylene	0.7357	19.01
Chlorobenzene	0.8558	19.73
Ethylbenzene	0.6243	20.20
m,p-Xylene	0.7367	20.41
Styrene	1.888	20.80
1,1,2,2-Tetrachloroethane	1.035	20.92
o-Xylene	0.7498	20.92
4-Ethyltoluene	0.6181	22.53
1,3,5-Trimethylbenzene	0.7088	22.65
1,2,4-Trimethylbenzene	0.7536	23.18
m-Dichlorobenzene	0.9643	23.31
Benzyl chloride	1.420	23.32
p-Dichlorobenzene	0.8912	23.41
o-Dichlorobenzene	1.004	23.88
1,2,4-Trichlorobenzene	2.150	26.71
Hexachlorobutadiene	0.4117	27.68

TABLE 6. GC-MS-SIM CALIBRATION TABLE

*** External Standard ***

Operator: JDF 8 Jan 87 10:02 am
Sample Info : SYR 1
Misc Info:
Integration File Name : DATA:SYR2A02A.I
Sequence Index: 1 Bottle Number : 2

Last Update: 8 Jan 87 8:13 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Peak Num	Type	Int Type	Ret Time	Signal Description			Compound Name	Area	Amount	
1		1	PP	5.020	Mass	85.00	amu	FREON 12	12893	4011 pptv	
2		1	PP	5.654	Mass	50.00	amu	METHYLCHLORI	4445	2586 pptv	
3		1	BP	6.525	Mass	85.00	amu	FREON 114	7067	1215 pptv	
4		1	PB	6.650	Mass	62.00	amu	VINYLCHLORID	2892	1929 pptv	*
5		1	BP	7.818	Mass	94.00	amu	METHYLBROMID	2401	1729 pptv	
6		1	BB	8.421	Mass	64.00	amu	ETHYLCHLORID	2134	2769 pptv	*
7		1	BV	9.940	Mass	101.00	amu	FREON 11	25069	6460 pptv	
8		1	BP	10.869	Mass	61.00	amu	VINDENECHLOR	5034	1700 pptv	
9		1	BP	11.167	Mass	49.00	amu	DICHLOROMETH	4803	2348 pptv	
10		1	PP	11.225	Mass	41.00	amu	ALLYLCHLORID	761	8247 pptv	*
11		1	BP	11.578	Mass	151.00	amu	3CHL3FLUETHA	5477	1672 pptv	
12		1	BP	12.492	Mass	63.00	amu	1,1DICHLOETH	5052	1738 pptv	*
13		1	VP	13.394	Mass	61.00	amu	c-1,2DICHLET	4761	1970 pptv	
14		1	PH	13.713	Mass	83.00	amu	CHLOROFORM	5327	1678 pptv	
15		1	BP	14.378	Mass	62.00	amu	1,2DICHLETHA	5009	2263 pptv	
16		1	PB	14.594	Mass	97.00	amu	METHCHLOROFO	6656	2334 pptv	
17		1	VP	15.009	Mass	78.00	amu	BENZENE	8352	2167 pptv	
18		1	VP	15.154	Mass	117.00	amu	CARBONTETRAC	5888	1915 pptv	
19		1	BB	15.821	Mass	63.00	amu	1,2DICHLPROP	3283	1799 pptv	*
20		1	BB	16.067	Mass	130.00	amu	TRICHLETHENE	4386	2109 pptv	
21		1	PB	16.941	Mass	75.00	amu	c-1,3DICHLPR	2226	987.3 pptv	
22		1	BP	17.475	Mass	75.00	amu	t-1,3DICHLPR	1626	689.2 pptv	
23		1	BB	17.594	Mass	97.00	amu	1,1,2CHLETHA	2721	1772 pptv	
24		1	BV	17.844	Mass	91.00	amu	TOLUENE	14417	2703 pptv	
25		1	PB	18.463	Mass	107.00	amu	EDB	4070	1365 pptv	*
26		1	PH	18.989	Mass	166.00	amu	TETRACHLETHE	6674	2065 pptv	
27		1	PB	19.705	Mass	112.00	amu	CHLOROBENZEN	5648	1524 pptv	
28		1	BP	20.168	Mass	91.00	amu	ETHYLBENZENE	11084	1842 pptv	
29		1	PB	20.372	Mass	91.00	amu	m,p-XYLENE	17989	3790 pptv	
30		1	BV	20.778	Mass	104.00	amu	STYRENE	3145	1695 pptv	
31		1	BH	20.867	Mass	83.00	amu	TETRACHLETHA	4531	1376 pptv	
32		1	BP	20.892	Mass	91.00	amu	o-XYLENE	9798	2010 pptv	
33		1	VV	22.488	Mass	105.00	amu	4-ETHYLTOLUE	7694	1481 pptv	
34		1	VB	22.609	Mass	105.00	amu	1,3,5METHBEN	6781	1705 pptv	
35		1	BB	23.144	Mass	105.00	amu	1,2,4METHBEN	7892	2095 pptv	
36		1	BV	23.273	Mass	146.00	amu	m-DICHLBENZE	3046	1119 pptv	
37		1	VV	23.279	Mass	91.00	amu	BENZYLCHLORI	3880	1006 pptv	
38		1	VB	23.378	Mass	146.00	amu	p-DICHLBENZE	6090	2164 pptv	
39		1	BP	23.850	Mass	146.00	amu	o-DICHLBENZE	2896	1249 pptv	
40		1	BB	26.673	Mass	180.00	amu	1,2,4CHLBENZ	562	767.1 pptv	
41		1	BB	27.637	Mass	225.00	amu	HEXACHLBUTAD	6309	1789 pptv	

TABLE 7. TYPICAL RETENTION TIME (MIN) AND CALIBRATION RESPONSE FACTORS (ppbv/area count) FOR TARGETED VOCs ASSOCIATED WITH FID AND ECD ANALYTICAL SYSTEM

Peak Number[1]	Compound	Retention Time (RT), minutes	FID Response Factor (RF) (ppbv/area count)	ECD Response Factor (ppbv/area count x 10^{-5})
1	Freon 12	3.65	3.465	13.89
2	Methyl chloride	4.30	0.693	
3	Freon 114	5.13	0.578	22.32
4	Vinyl chloride	5.28	0.406	
5	Methyl bromide	6.44		26.34
6	Ethyl chloride	7.06	0.413	
7	Freon 11	8.60	6.367	1.367
8	Vinylidene chloride	9.51	0.347	
9	Dichloromethane	9.84	0.903	
10	Trichlorotrifluoroethane	10.22	0.374	3.955
11	1,1-Dichloroethane	11.10	0.359	
12	cis-1,2-Dichloroethylene	11.99	0.368	
13	Chloroform	12.30	1.059	11.14
14	1,2-Dichloroethane	12.92	0.409	
15	Methyl chloroform	13.12	0.325	3.258
16	Benzene	13.51	0.117	
17	Carbon tetrachloride	13.64	1.451	1.077
18	1,2-Dichloropropane	14.26	0.214	
19	Trichloroethylene	14.50	0.327	8.910
20	cis-1,3-Dichloropropene	15.31		
21	trans-1,3-Dichloropropene	15.83		
22	1,1,2-Trichloroethane	15.93	0.336	
23	Toluene	16.17	0.092	
24	1,2-Dibromoethane (EDB)	16.78	0.366	5.137
25	Tetrachloroethylene	17.31	0.324	1.449
26	Chlorobenzene	18.03	0.120	
27	Ethylbenzene	18.51	0.092	
28	m,p-Xylene	18.72	0.095	
29	Styrene	19.12	0.143	
30	1,1,2,2-Tetrachloroethane	19.20		9.856
31	o-Xylene	19.23		
32	4-Ethyltoluene	20.82	0.100	
33	1,3,5-Trimethylbenzene	20.94	0.109	
34	1,2,4-Trimethylbenzene	21.46	0.111	
35	m-Dichlorobenzene	21.50		
36	Benzyl chloride	21.56		
37	p-Dichlorobenzene	21.67	0.188	
38	o-Dichlorobenzene	22.12	0.188	
39	1,2,4-Trichlorobenzene	24.88	0.667	
40	Hexachlorobutadiene	25.82	0.305	1.055

[1] Refer to Figures 15 and 16 for peak location

TABLE 8. TYPICAL RETENTION TIME (minutes) FOR SELECTED ORGANICS USING GC-FID-ECD-PID* ANALYTICAL SYSTEM

Compound	Retention-Time (minutes)		
	FID	ECD	PID
Acetylene	2.984	--	--
1,3-Butadiene	3.599	--	3.594
Vinyl chloride	3.790	--	3.781
Chloromethane	5.137	--	--
Chloroethane	5.738	--	--
Bromoethane	8.154	--	--
Methylene Chloride	9.232	--	9.218
trans-1,2-Dichloroethylene	10.077	--	10.065
1,1-Dichloroethane	11.190	--	--
Chloroprene	11.502	--	11.491
Perfluorobenzene	13.077	13.078	13.069
Bromochloromethane	13.397	13.396	13.403
Chloroform	13.768	13.767	13.771
1,1,1-Trichloroethane	14.151	14.153	14.158
Carbon Tetrachloride	14.642	14.667	14.686
Benzene/1,2-Dichloroethane	15.128	--	15.114
Perfluorotoluene	15.420	15.425	15.412
Trichloroethylene	17.022	17.024	17.014
1,2-Dichloropropene	17.491	17.805	17.522
Bromodichloromethane	18.369	--	--
trans-1,3-Dichloropropylene	19.694	19.693	19.688
Toluene	20.658	--	20.653
cis-1,3-Dichloropropylene	21.461	21.357	21.357
1,1,2-Trichloroethane	21.823	--	--
Tetrachloroethylene	22.340	22.346	22.335
Dibromochloromethane	22.955	22.959	22.952
Chlorobenzene	24.866	--	24.861
m/p-Xylene	25.763	--	25.757
Styrene/o-Xylene	27.036	--	27.030
Bromofluorobenzene	28.665	28.663	28.660
1,1,2,2-Tetrachloroethane	29.225	29.227	29.228
m-Dichlorobenzene	32.347	32.345	32.342
p-Dichlorobenzene	32.671	32.669	32.666
o-Dichlorobenzene	33.885	33.883	33.880

* Varian® 3700 GC equipped with J & W Megabore® DB 624 Capillary Column (30 m X 0.53 I.D. mm) using helium carrier gas.

TABLE 9. GC-MS-SIM CALIBRATION TABLE

Last Update: 18 Dec 86 7:54 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.000

Ret Time	Pk#	Signal	Descr	Amt pptv	Lvl	[Area]	Pk-Type	Partial Name
5.008	1	Mass	85.00 amu	13620	1	72974	1	FREON 12
5.690	2	Mass	50.00 amu	12720	1	36447	1	METHYLCHLORID
6.552	3	Mass	85.00 amu	8380	1	81251	1	FREON 114
6.709	4	Mass	62.00 amu	8050	1	20118	1	VINYLCHLORIDE
7.831	5	Mass	94.00 amu	12210	1	28265	1	METHYLBROMIDE
8.431	6	Mass	64.00 amu	12574	1	16149	1	ETHYLCHLORIDE
9.970	7	Mass	101.00 amu	12380	1	80088	1	FREON 11
10.927	8	Mass	61.00 amu	7890	1	38954	1	VINDENECHLORI
11.209	9	Mass	49.00 amu	12760	1	43507	1	DICHLOROMETHA
11.331	10	Mass	41.00 amu	12650	1	1945	1	ALLYLCHLORIDE
11.595	11	Mass	151.00 amu	7420	1	40530	1	3CHL3FLUETHAN
12.502	12	Mass	63.00 amu	12710	1	61595	1	1,1DICHLOETHA
13.403	13	Mass	61.00 amu	12630	1	50900	1	c-1,2DICHLETH
13.747	14	Mass	83.00 amu	7670	1	40585	1	CHLOROFORM
14.387	15	Mass	62.00 amu	9040	1	33356	1	1,2DICHLETHAN
14.623	16	Mass	97.00 amu	8100	1	38503	1	METHCHLOROFOR
15.038	17	Mass	78.00 amu	10760	1	69119	1	BENZENE
15.183	18	Mass	117.00 amu	8340	1	42737	1	CARBONTETRACH
15.829	19	Mass	63.00 amu	12780	1	38875	1	1,2DICHLPROPA
16.096	20	Mass	130.00 amu	8750	1	30331	1	TRICHLETHENE
16.956	21	Mass	75.00 amu	4540	1	17078	1	c-1,3DICHLPRO
17.492	22	Mass	75.00 amu	3380	1	13294	1	t-1,3DICHLPRO
17.610	23	Mass	97.00 amu	12690	1	32480	1	1,1,2CHLETHAN
17.862	24	Mass	91.00 amu	10010	1	86036	1	TOLUENE
18.485	25	Mass	107.00 amu	6710	1	33350	1	EDB
19.012	26	Mass	166.00 amu	7830	1	43454	1	TETRACHLETHEN
19.729	27	Mass	112.00 amu	7160	1	44224	1	CHLOROBENZENE
20.195	28	Mass	91.00 amu	12740	1	127767	1	ETHYLBENZENE
20.407	29	Mass	91.00 amu	25400	1	200973	1	m,p-XYLENE
20.806	30	Mass	104.00 amu	12390	1	38332	1	STYRENE
20.916	31	Mass	83.00 amu	11690	1	64162	1	TETRACHLETHAN
20.921	32	Mass	91.00 amu	11085	1	90096	1	o-XYLENE
22.528	33	Mass	105.00 amu	12560	1	108747	1	4-ETHYLTOLUEN
22.648	34	Mass	105.00 amu	12620	1	83666	1	1,3,5METHBENZ
23.179	35	Mass	105.00 amu	12710	1	79833	1	1,2,4METHBENZ
23.307	36	Mass	146.00 amu	12650	1	57409	1	m-DICHLBENZEN
23.317	37	Mass	91.00 amu	7900	1	50774	1	BENZYLCHLORID
23.413	38	Mass	146.00 amu	12390	1	58127	1	p-DICHLBENZEN
23.885	39	Mass	146.00 amu	13510	1	52233	1	o-DICHLBENZEN
26.714	40	Mass	180.00 amu	15520	1	18967	1	1,2,4CHLBENZE
27.680	41	Mass	225.00 amu	7470	1	43920	1	HEXACHLBUTADI

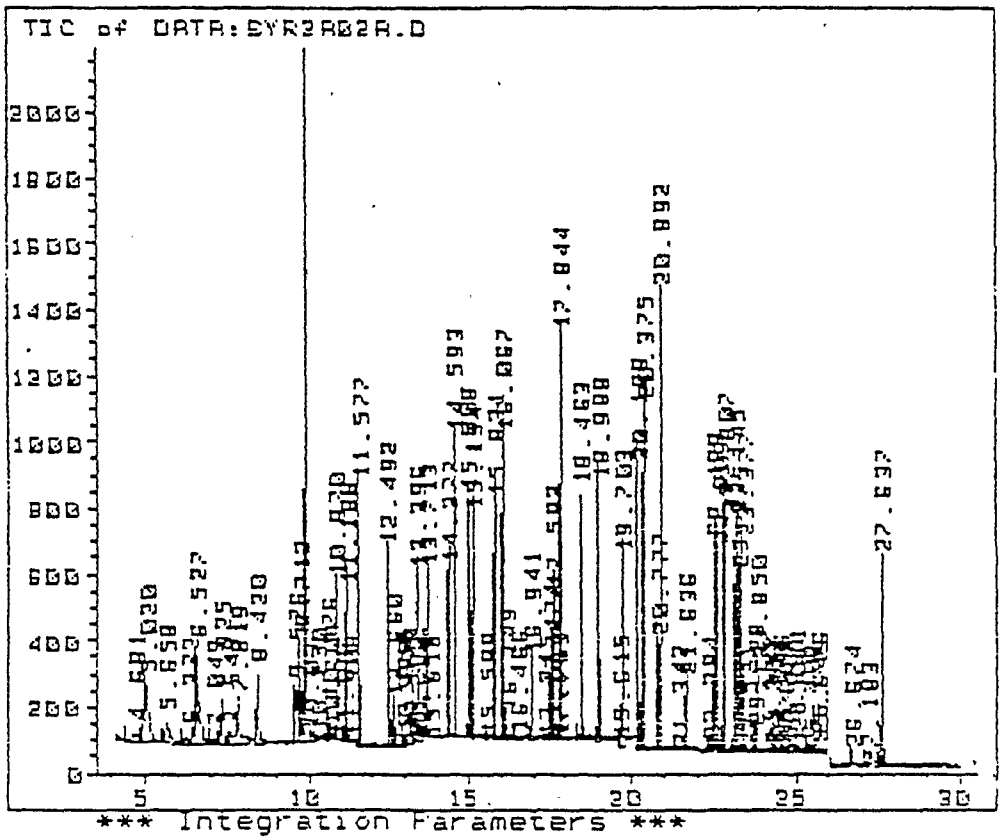
TABLE 10. EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS

Data file: DATA:SYR2A02A.D
File type: GC / MS DATA FILE

Name Info: SYR 1
Misc Info:
Operator : JDF

Date : 8 Jan 87 10:02 am
Instrment: MS_5970
Inlet : GC

Sequence index : 1
Als bottle num : 2
Replicate num : 1



*** Integration Parameters ***

FALSE : Shoulder Detection Enabled
0.020 : Expected Peak Width (Min)
11 : Initial Peak Detection Threshold

4.000 THRESHOLD 5.000
4.000 PEAK_WIDTH 0.200
9.800 PEAK_WIDTH 0.060

AR303827

TABLE 10. EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS (cont.)

Operator: JDF 8 Jan 87 10:0[illegible] am
Sample Info : SYR 1
Misc Info:
Integration File Name : DATA:SYR2A02A.I
Sequence Index: 1 Bottle Number : 2

Last Update: 8 Jan 87 8:13 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Peak Num	Type	Int Type	Ret Time	Signal Description		Compound Name	Area	Amount	
1		1 PP	5.020	Mass	85.00 amu	FREON 12	12893	4011 pptv	
2		1 PP	5.654	Mass	50.00 amu	METHYLCHLORI	4445	2586 pptv	
3		1 BP	6.525	Mass	85.00 amu	FREON 114	7067	1215 pptv	
4		1 PB	6.650	Mass	62.00 amu	VINYLCHLORID	2892	1929 pptv	*
5		1 BP	7.818	Mass	94.00 amu	METHYLBROMID	2401	1729 pptv	
6		1 BB	8.421	Mass	64.00 amu	ETHYLCHLORID	2134	2769 pptv	*
7		1 BV	9.940	Mass	101.00 amu	FREON 11	25069	6460 pptv	
8		1 BP	10.869	Mass	61.00 amu	VINDENECHLOR	5034	1700 pptv	
9		1 BP	11.187	Mass	49.00 amu	DICHLOROMETH	4803	2348 pptv	
10		1 PP	11.225	Mass	41.00 amu	ALLYLCHLORID	761	8247 pptv	*
11		1 BP	11.578	Mass	151.00 amu	3CHL3FLUETHA	5477	1672 pptv	
12		1 BP	12.492	Mass	63.00 amu	1,1DICHLOETH	5052	1738 pptv	*
13		1 VP	13.394	Mass	61.00 amu	c-1,2DICHLET	4761	1970 pptv	
14		1 PH	13.713	Mass	83.00 amu	CHLOROFORM	5327	1678 pptv	
15		1 BP	14.378	Mass	62.00 amu	1,2DICHLETHA	5009	2263 pptv	
16		1 PB	14.594	Mass	97.00 amu	METHCHLOROFO	6656	2334 pptv	
17		1 VP	15.009	Mass	78.00 amu	BENZENE	8352	2167 pptv	
18		1 VP	15.154	Mass	117.00 amu	CARBONTETRAC	5888	1915 pptv	
19		1 BB	15.821	Mass	63.00 amu	1,2DICHLPROP	3283	1799 pptv	*
20		1 BB	16.067	Mass	130.00 amu	TRICHLETHENE	4386	2109 pptv	
21		1 PB	16.941	Mass	75.00 amu	c-1,3DICHLPR	2228	987.3 pptv	
22		1 BP	17.475	Mass	75.00 amu	t-1,3DICHLPR	1626	689.2 pptv	
23		1 BB	17.594	Mass	97.00 amu	1,1,2CHLETHA	2721	1772 pptv	
24		1 BV	17.844	Mass	91.00 amu	TOLUENE	14417	2733 pptv	
25		1 PB	18.463	Mass	107.00 amu	EDB	4070	1365 pptv	*
26		1 PH	18.989	Mass	166.00 amu	TETRACHLETHE	6874	2065 pptv	
27		1 PB	19.705	Mass	112.00 amu	CHLOROBENZEN	5648	1524 pptv	
28		1 BP	20.168	Mass	91.00 amu	ETHYLBENZENE	11084	1842 pptv	
29		1 PB	20.372	Mass	91.00 amu	m,p-XYLENE	17989	3790 pptv	
30		1 BV	20.778	Mass	104.00 amu	STYRENE	3145	1695 pptv	
31		1 BH	20.887	Mass	83.00 amu	TETRACHLETHA	4531	1376 pptv	
32		1 BP	20.892	Mass	91.00 amu	o-XYLENE	9798	2010 pptv	
33		1 VV	22.488	Mass	105.00 amu	4-ETHYLTOLUE	7694	1481 pptv	
34		1 VB	22.609	Mass	105.00 amu	1,3,5METHBEN	6781	1705 pptv	
35		1 BB	23.144	Mass	105.00 amu	1,2,4METHBEN	7892	2095 pptv	
36		1 BV	23.273	Mass	146.00 amu	m-DICHLBENZE	3046	1119 pptv	
37		1 VV	23.279	Mass	91.00 amu	BENZYLCHLORI	3880	1006 pptv	
38		1 VB	23.378	Mass	146.00 amu	p-DICHLBENZE	6090	2164 pptv	
39		1 BP	23.850	Mass	146.00 amu	o-DICHLBENZE	2896	1249 pptv	
40		1 BB	26.673	Mass	180.00 amu	1,2,4CHLBENZ	562	767.1 pptv	
41		1 BB	27.637	Mass	225.00 amu	HEXACHLBUTAD	6309	1789 pptv	

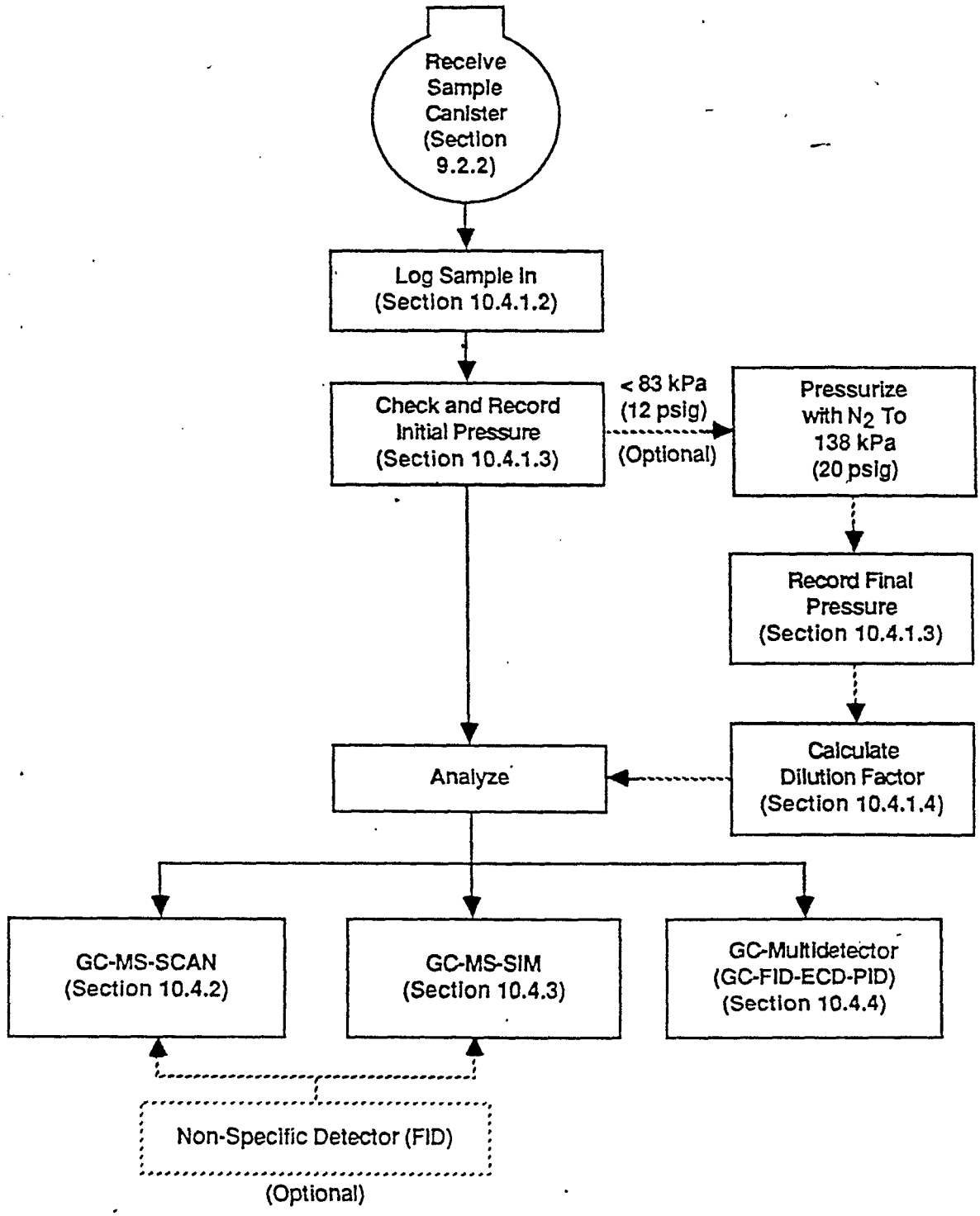
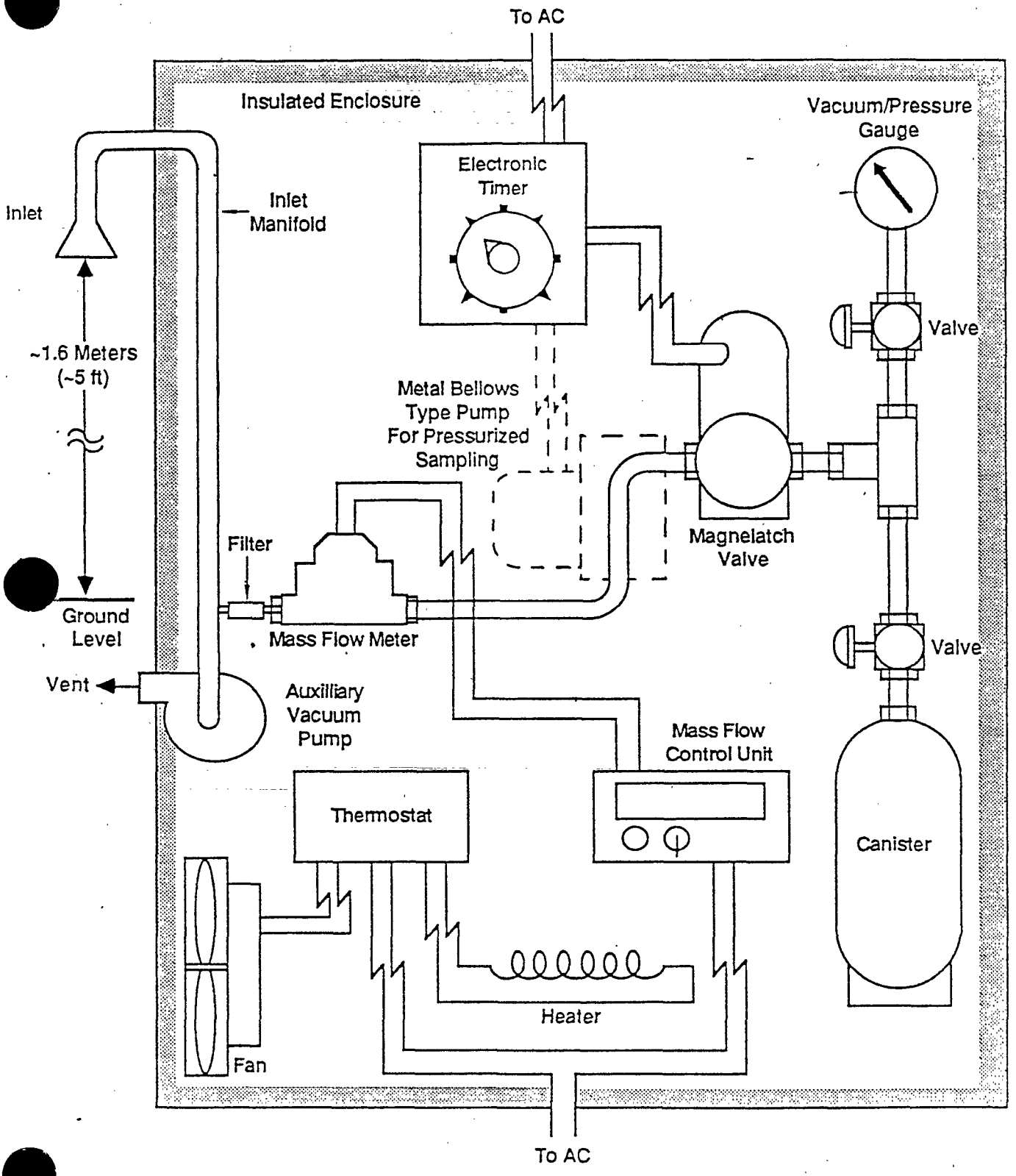


FIGURE 1. ANALYTICAL SYSTEMS AVAILABLE FOR CANISTER VOC IDENTIFICATION AND QUANTITATION

T014-75



AR303830

AR303830

FIGURE 2. SAMPLER CONFIGURATION FOR SUBATMOSPHERIC PRESSURE OR PRESSURIZED CANISTER SAMPLING

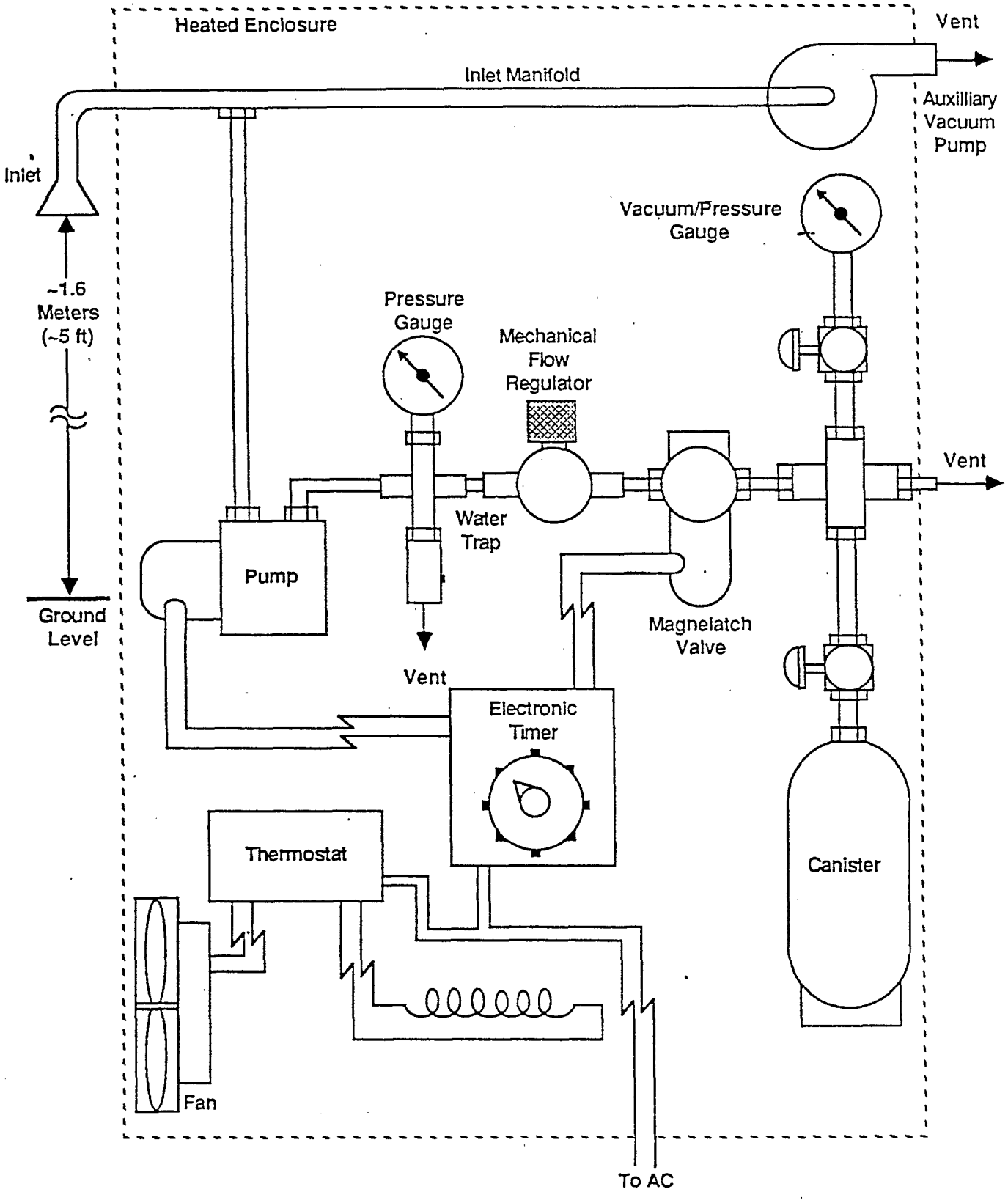


FIGURE 3. ALTERNATIVE SAMPLER CONFIGURATION FOR PRESSURIZED CANISTER SAMPLING

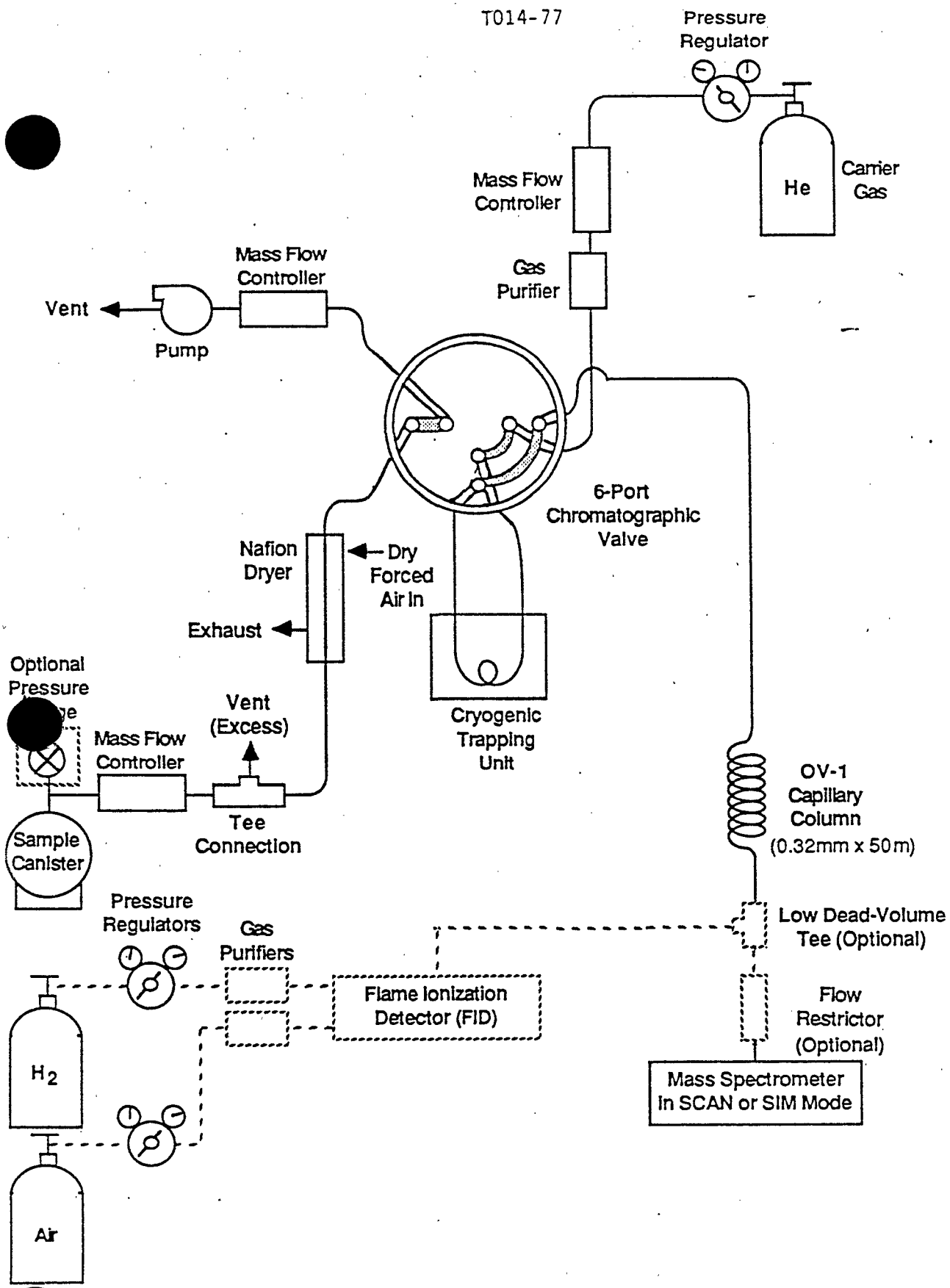


FIGURE 4. CANISTER ANALYSIS UTILIZING GC-MS-SCAN-SIM ANALYTICAL SYSTEM WITH OPTIONAL FLAME IONIZATION DETECTOR WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN THE SAMPLE DESORPTION MODE

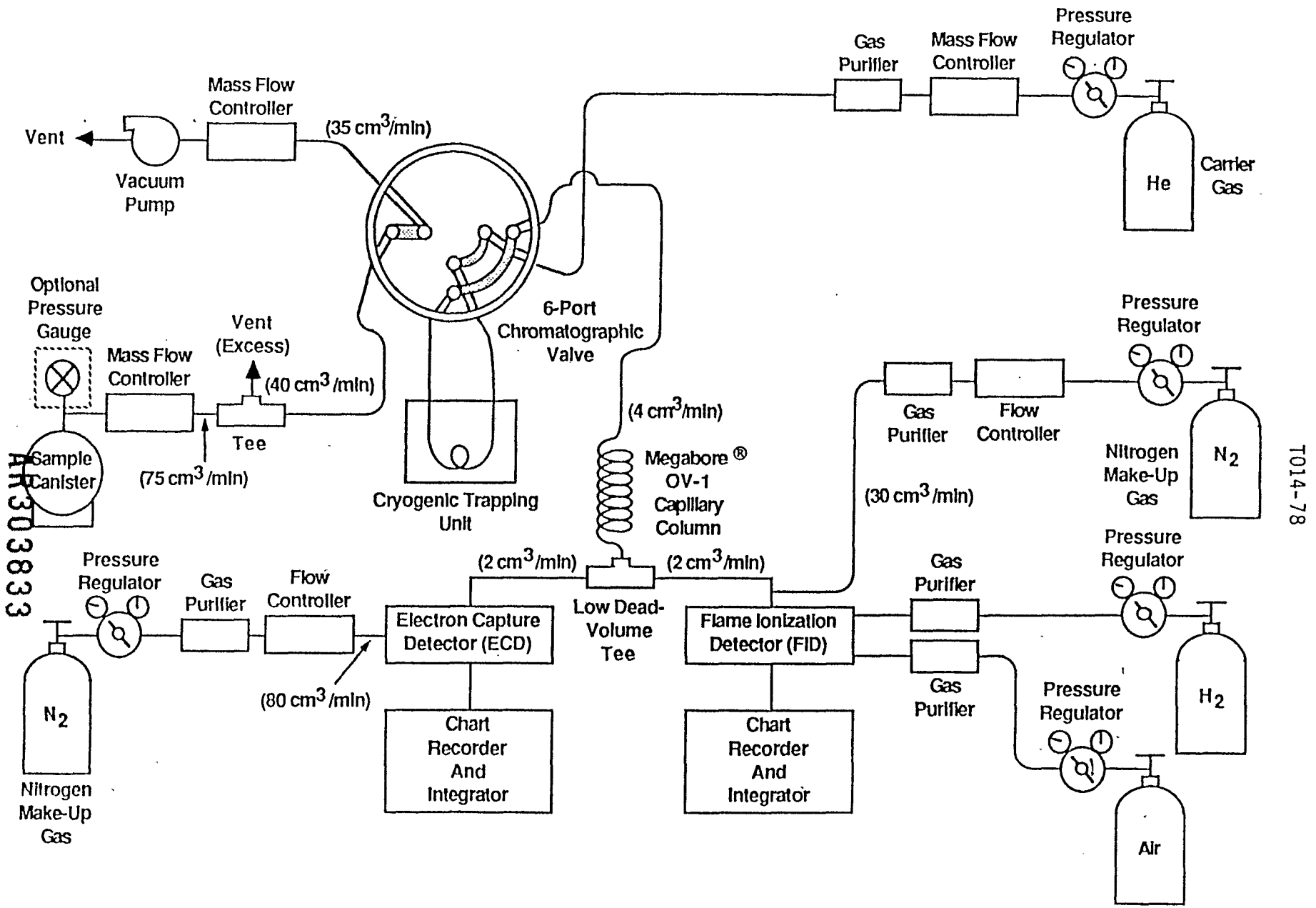


FIGURE 5. GC-FID-ECD ANALYTICAL SYSTEM WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN THE SAMPLE DESORPTION MODE

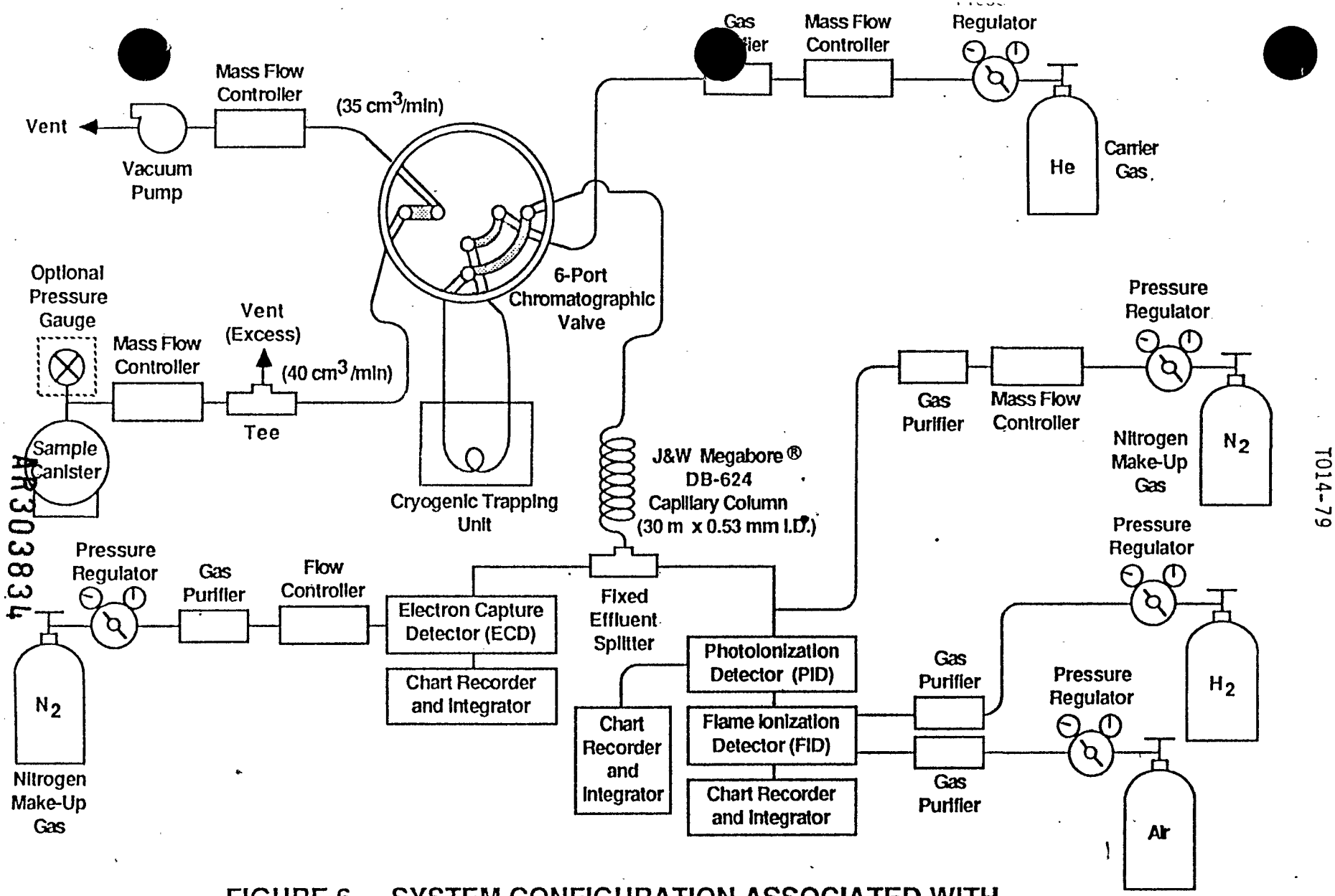
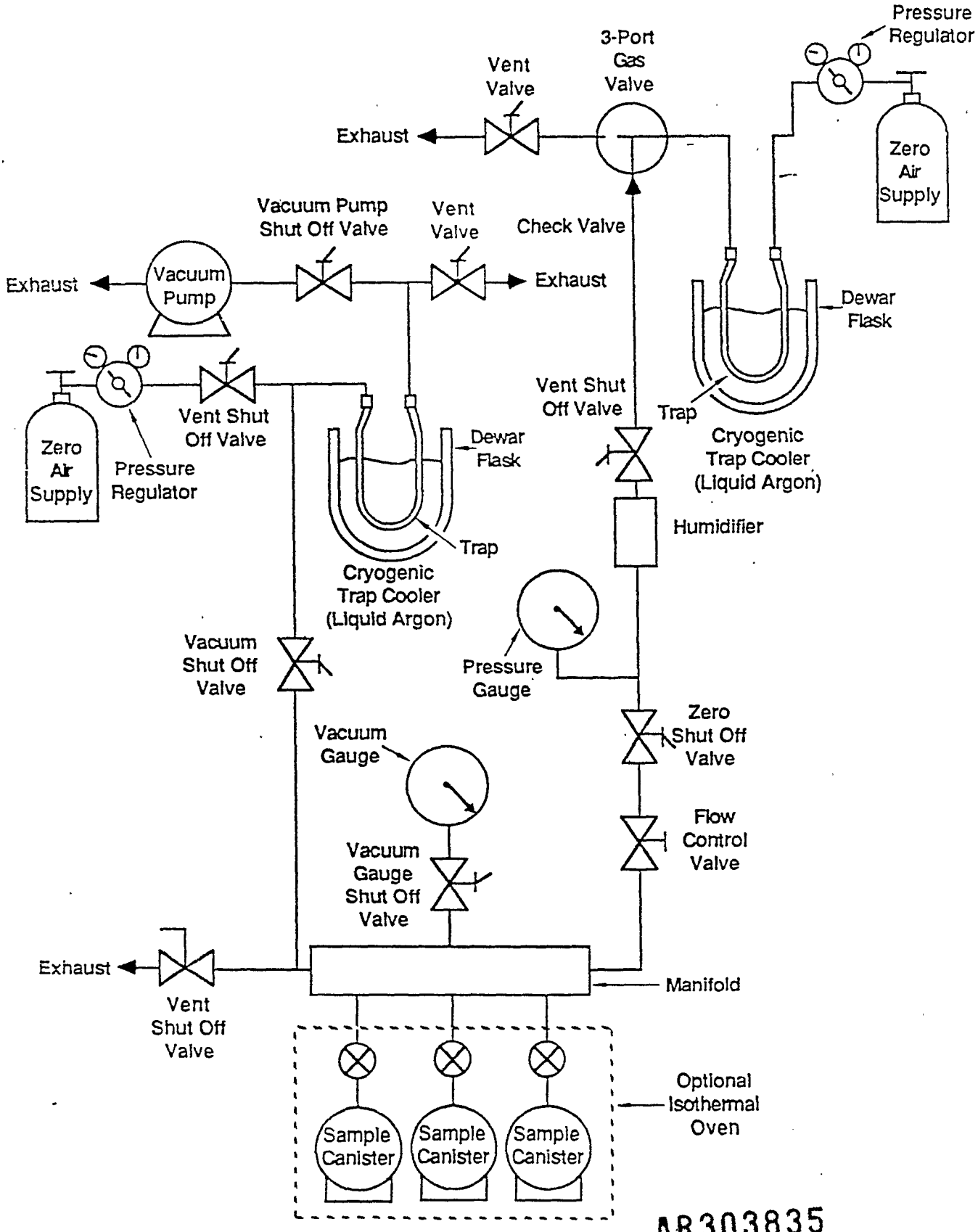


FIGURE 6. SYSTEM CONFIGURATION ASSOCIATED WITH THE GC-FID-ECD-PID ANALYTICAL SYSTEM WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN THE SAMPLE DESORPTION MODE



AR303835

FIGURE 7. CANISTER CLEANING SYSTEM

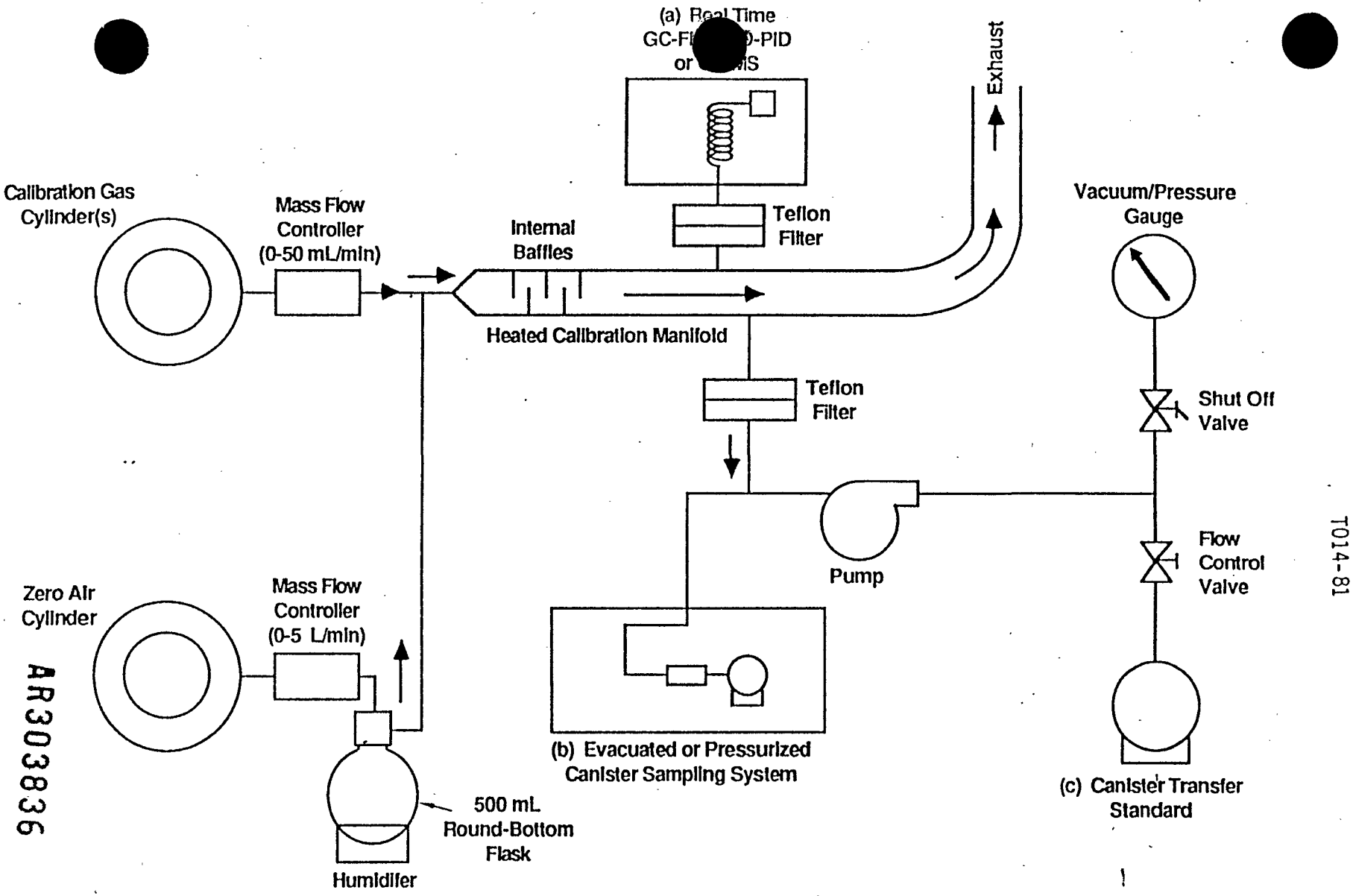
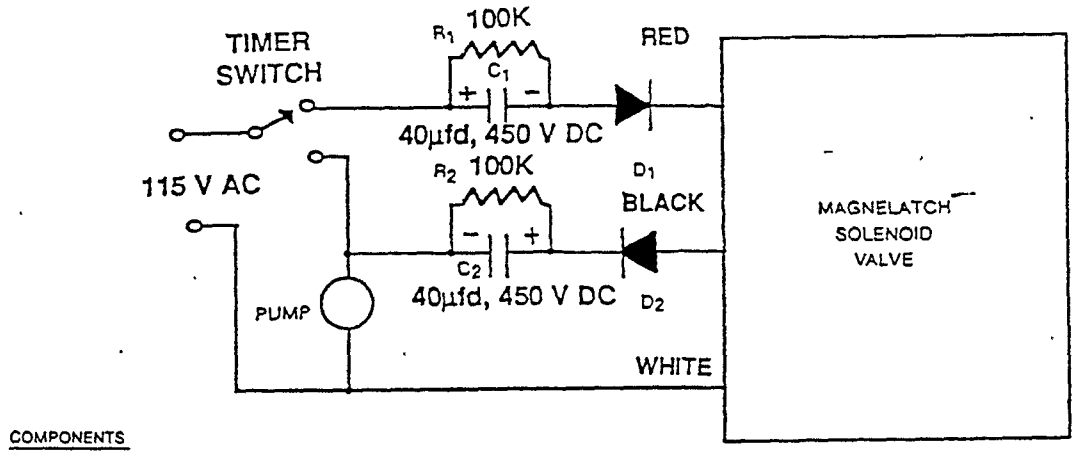


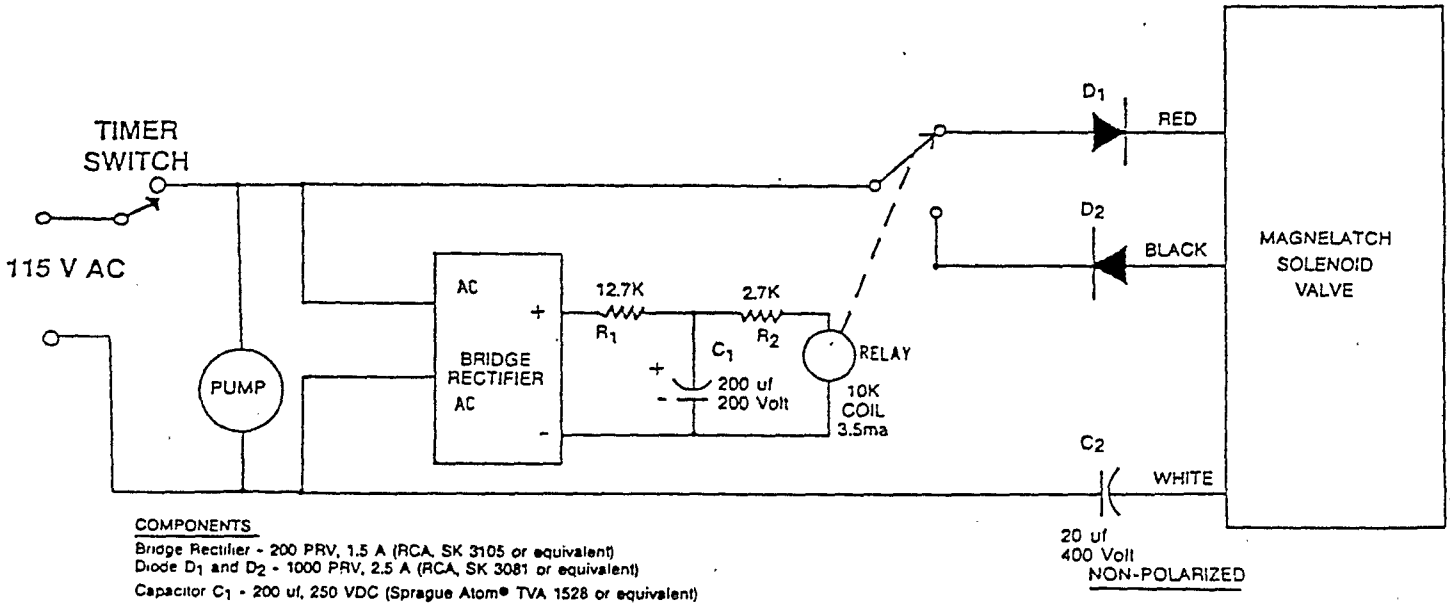
FIGURE 8. SCHEMATIC OF CALIBRATION SYSTEM AND MANIFOLD FOR (a) ANALYTICAL SYSTEM CALIBRATION, (b) TESTING CANISTER SAMPLING SYSTEM AND (c) PREPARING CANISTER TRANSFER STANDARDS.



COMPONENTS

Capacitor C_1 and C_2 - 40 uf, 450 VDC (Sprague Atom® TVA 1712 or equivalent)
Resister R_1 and R_2 - 0.5 watt, 5% tolerance
Diode D_1 and D_2 - 1000 PRV, 2.5 A (RCA, SK 3081 or equivalent)

(a). Simple Circuit For Operating Magnelatch Valve



COMPONENTS

Bridge Rectifier - 200 PRV, 1.5 A (RCA, SK 3105 or equivalent)
Diode D_1 and D_2 - 1000 PRV, 2.5 A (RCA, SK 3081 or equivalent)
Capacitor C_1 - 200 uf, 250 VDC (Sprague Atom® TVA 1528 or equivalent)
Capacitor C_2 - 20 uf, 400 VDC Non-Polarized (Sprague Atom® TVAN 1652 or equivalent)
Relay - 10,000 ohm coil, 3.5 ma (AMF Potter and Brumfield, KCP 5, or equivalent)
Resister R_1 and R_2 - 0.5 watt, 5% tolerance

(b). Improved Circuit Designed To Handle Power Interruptions

FIGURE 9. ELECTRICAL PULSE CIRCUITS FOR DRIVING SKINNER MAGNELATCH SOLENOID VALVE WITH A MECHANICAL TIMER

T014-83

CANISTER SAMPLING FIELD DATA SHEET

A. GENERAL INFORMATION

SITE LOCATION: _____
SITE ADDRESS: _____
_____
_____
SAMPLING DATE: _____

SHIPPING DATE: _____
CANISTER SERIAL NO.: _____
SAMPLER ID: _____
OPERATOR: _____
CANISTER LEAK CHECK DATE: _____

B. SAMPLING INFORMATION

	TEMPERATURE				PRESSURE	
	INTERIOR	AMBIENT	MAXIMUM	MINIMUM	CANISTER PRESSURE	
START			X	X		
STOP					X	

	SAMPLING TIMES		FLOW RATES		
	LOCAL TIME	ELAPSED TIME METER READING	MANIFOLD FLOW RATE	CANISTER FLOW RATE	FLOW CONTROLLER READOUT
START					
STOP					

SAMPLING SYSTEM CERTIFICATION DATE: _____
QUARTERLY RECERTIFICATION DATE: _____

C. LABORATORY INFORMATION

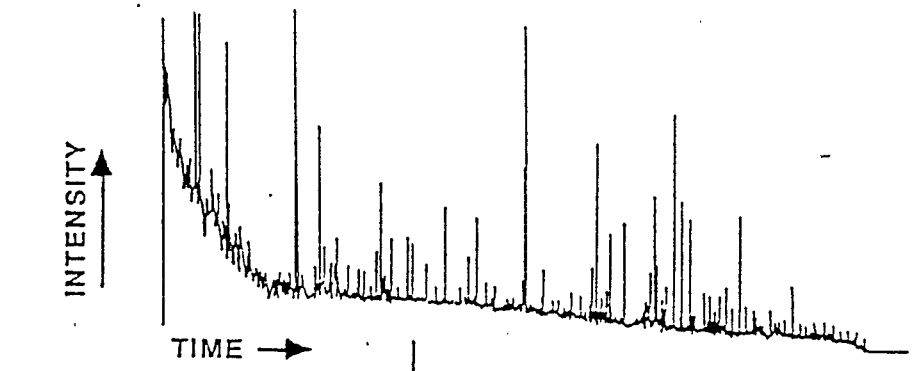
DATE RECEIVED: _____
RECEIVED BY: _____
INITIAL PRESSURE: _____
FINAL PRESSURE: _____
DILUTION FACTOR: _____
ANALYSIS
- GC-FID-ECD DATE: _____
- GC-MSD-SCAN DATE: _____
- GC-MSD-SIM DATE: _____

RESULTS*: _____
_____
- GC-FID-ECD: _____
- GC-MSD-SCAN: _____
- GC-MSD-SIM: _____

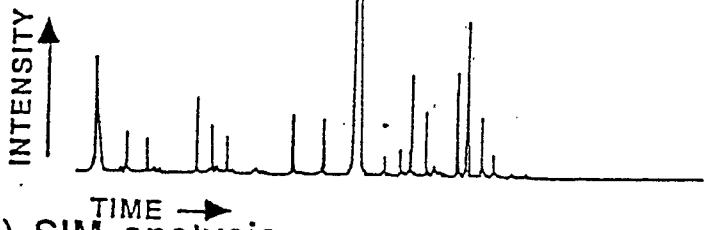
_____
SIGNATURE/TITLE

* ATTACH DATA SHEETS

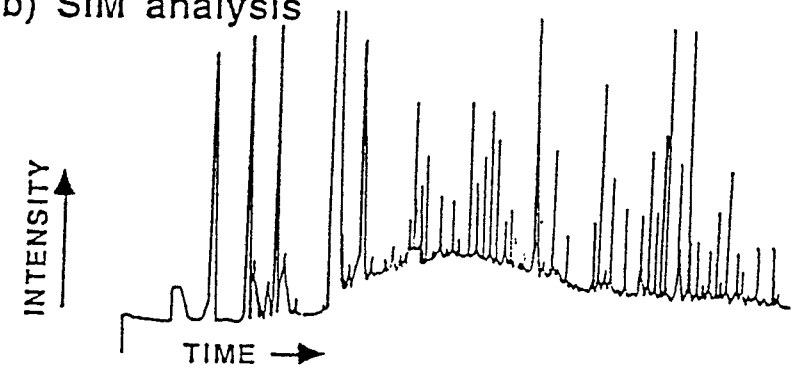
FIGURE 10. CANISTER SAMPLING FIELD DATA SHEET



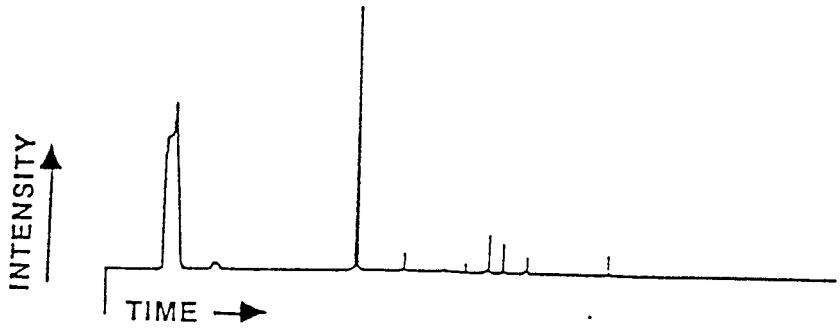
(a) SCAN analysis



(b) SIM analysis



(c) FID analysis



(d) ECD analysis

FIGURE 11. TYPICAL CHROMATOGRAMS OF A VOC SAMPLE ANALYZED BY GC-MS-SCAN-SIM MODE AND GC-MULTIDETECTOR MODE

AR303839

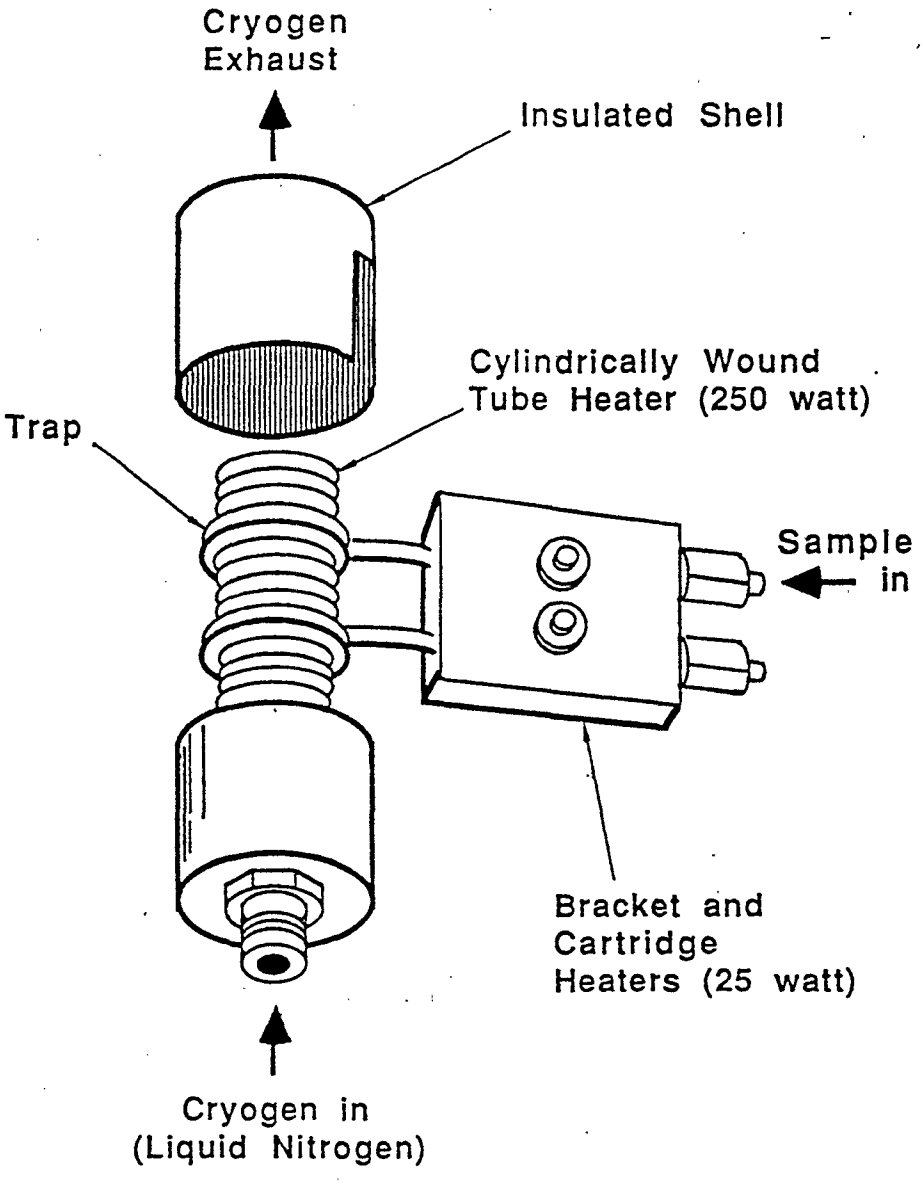


FIGURE 12. CRYOGENIC TRAPPING UNIT

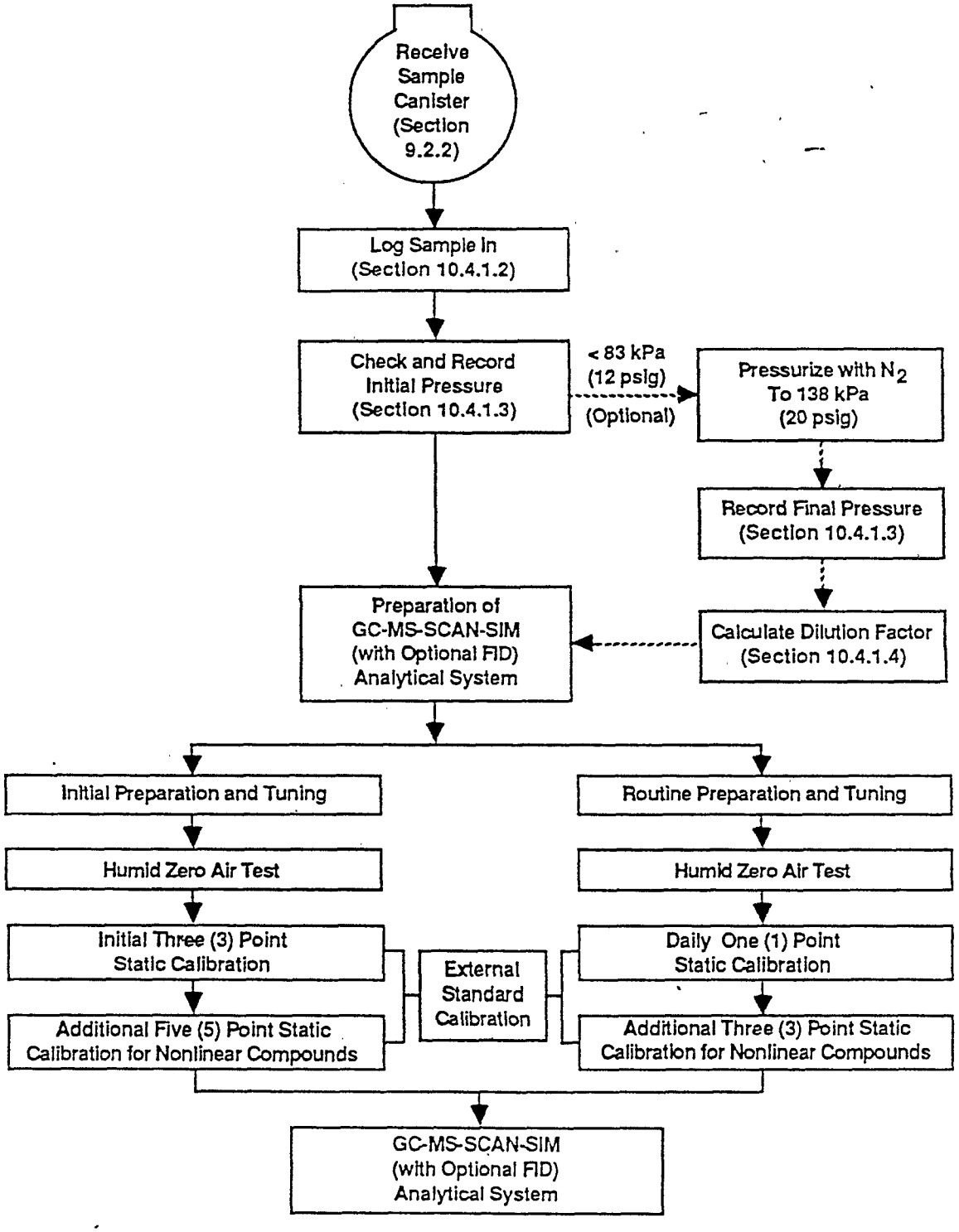


FIGURE 13. FLOWCHART OF GC-MS-SCAN-SIM ANALYTICAL SYSTEM PREPARATION (WITH OPTIONAL FID SYSTEM)

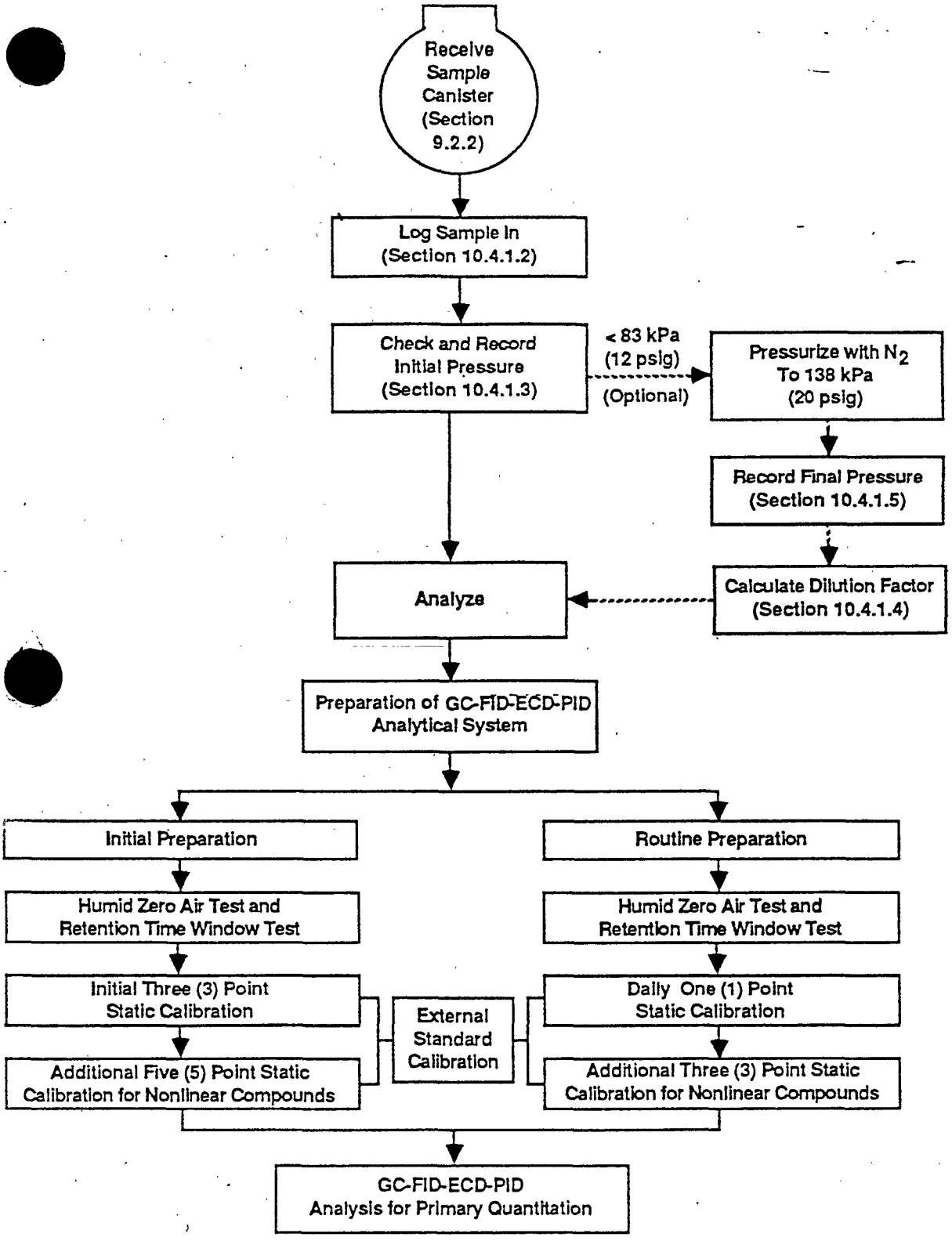
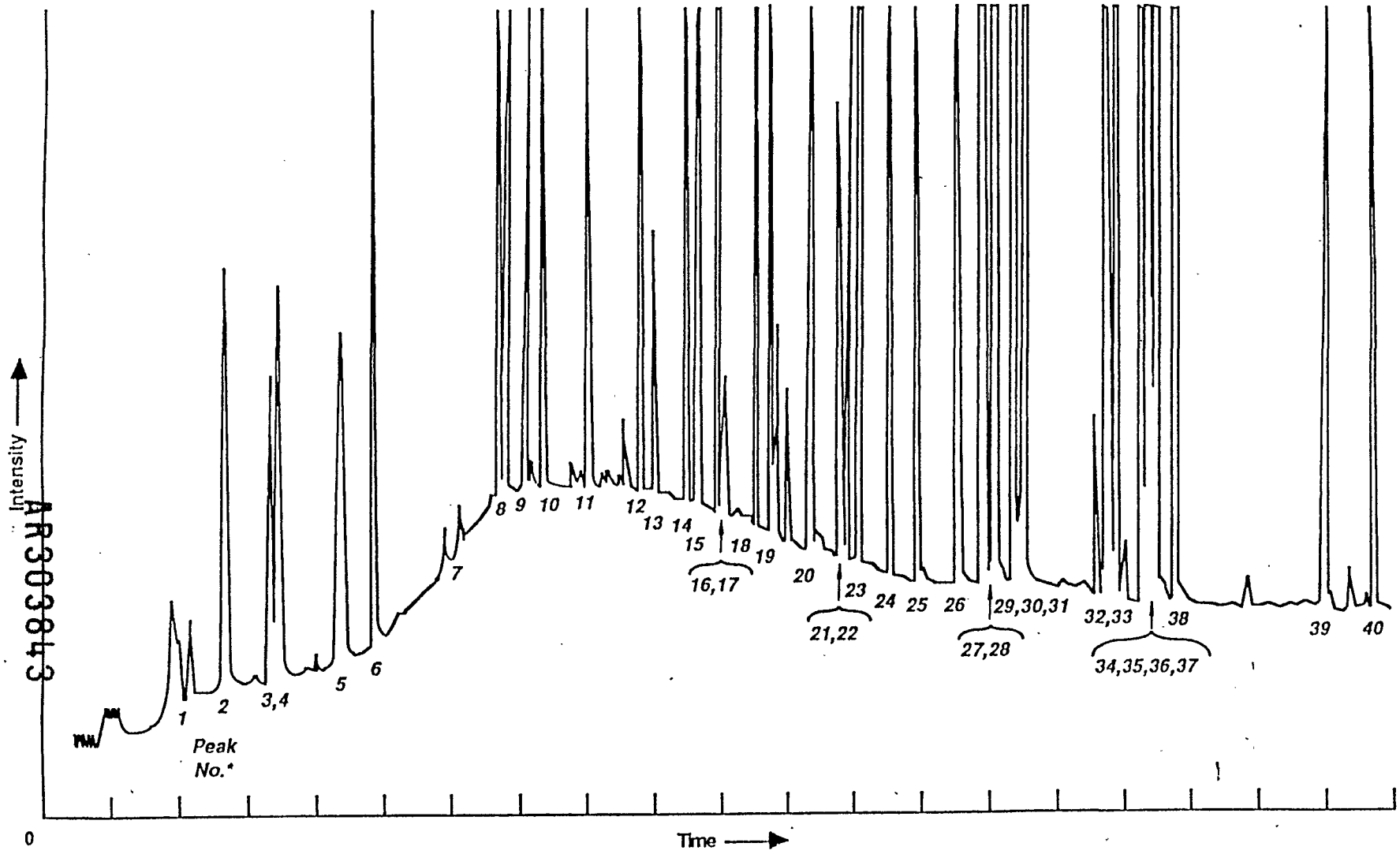
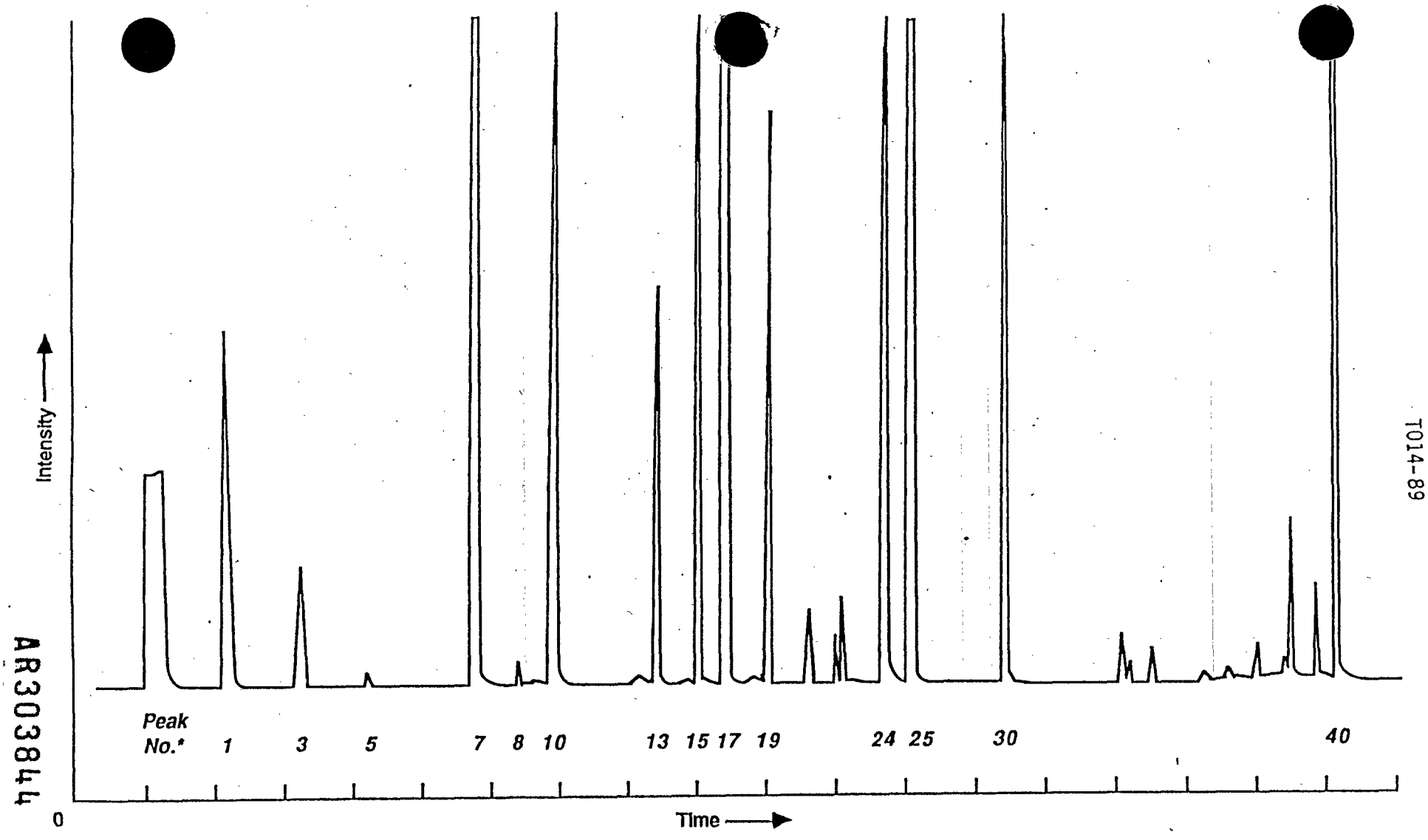


FIGURE 14. FLOWCHART OF GC-FID-ECD-PID ANALYTICAL SYSTEM PREPARATION



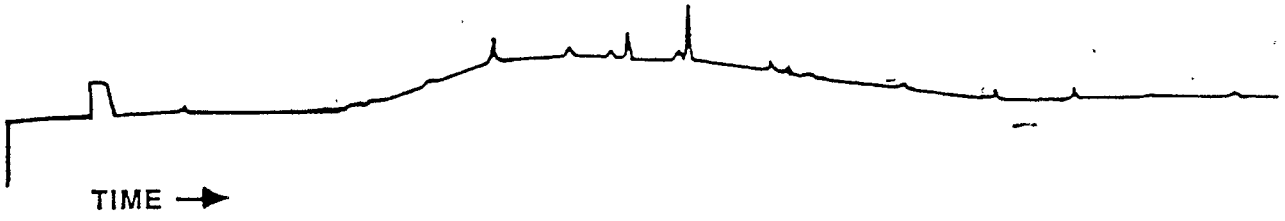
* See Table 7.0 For Peak Identification

FIGURE 15. TYPICAL FID RESPONSE TO SELECTIVE VOCs

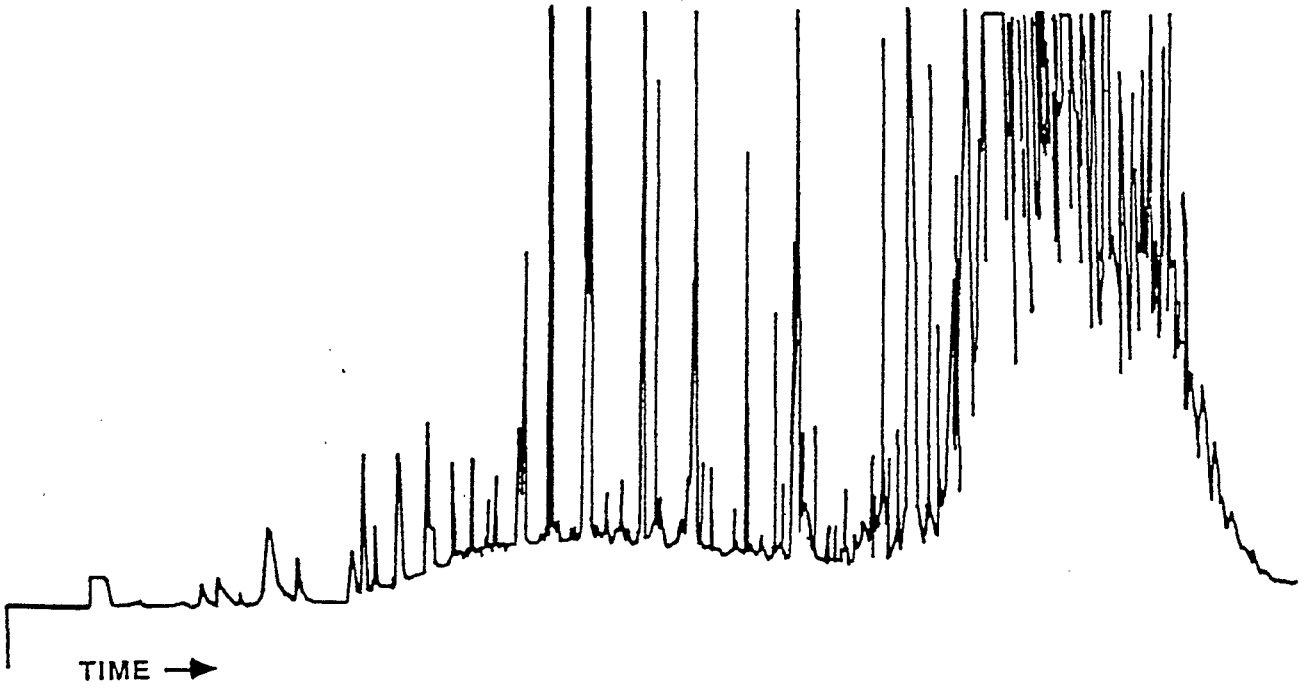


* See Table 7.0 For Peak Identification

FIGURE 16. TYPICAL ECD RESPONSE TO SELECTIVE VOCs



(a). Certified Sampler



(b). Contaminated Sampler

FIGURE 17. EXAMPLE OF HUMID ZERO AIR TEST RESULTS FOR A CLEAN SAMPLER (a) AND A CONTAMINATED SAMPLER (

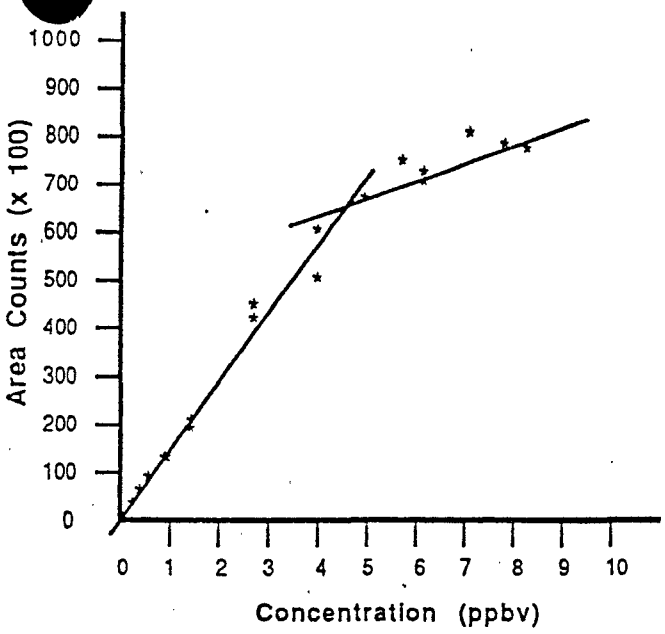


FIGURE 18(a). NONLINEAR RESPONSE OF TETRACHLOROETHYLENE ON THE ECD

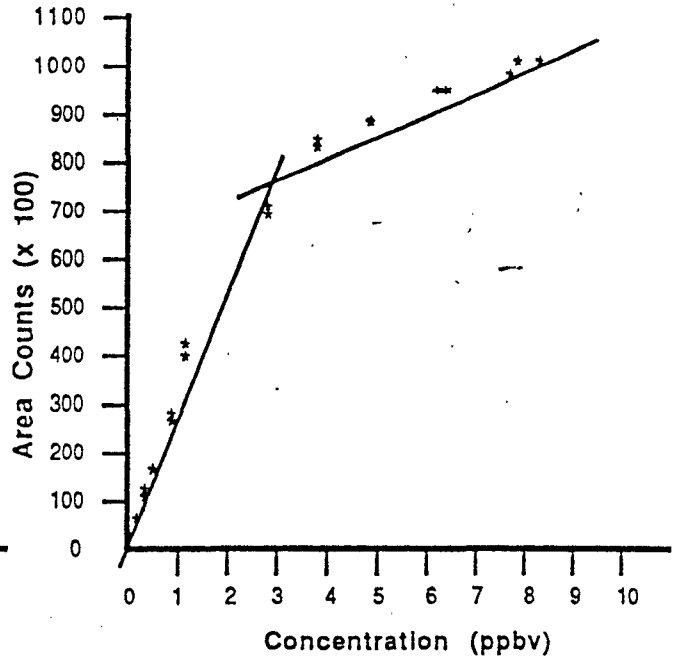


FIGURE 18(b). NONLINEAR RESPONSE OF CARBON TETRACHLORIDE ON THE ECD

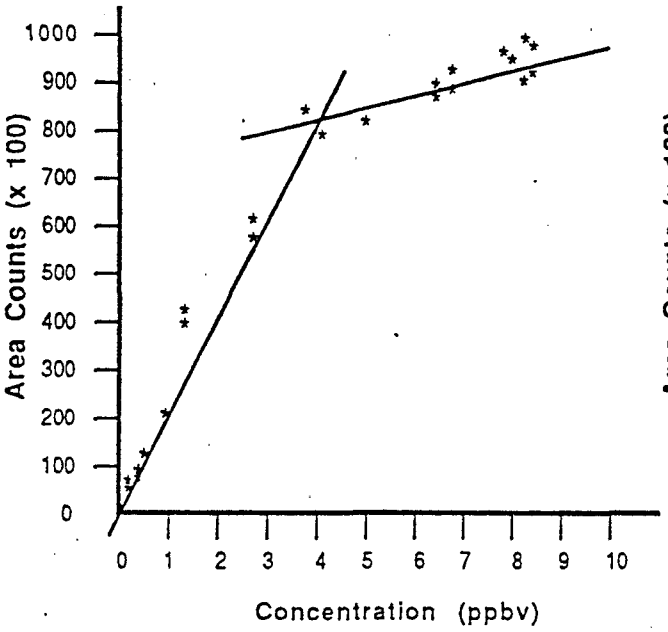


FIGURE 18(c). NONLINEAR RESPONSE OF HEXACHLOROBUTADIENE ON THE ECD

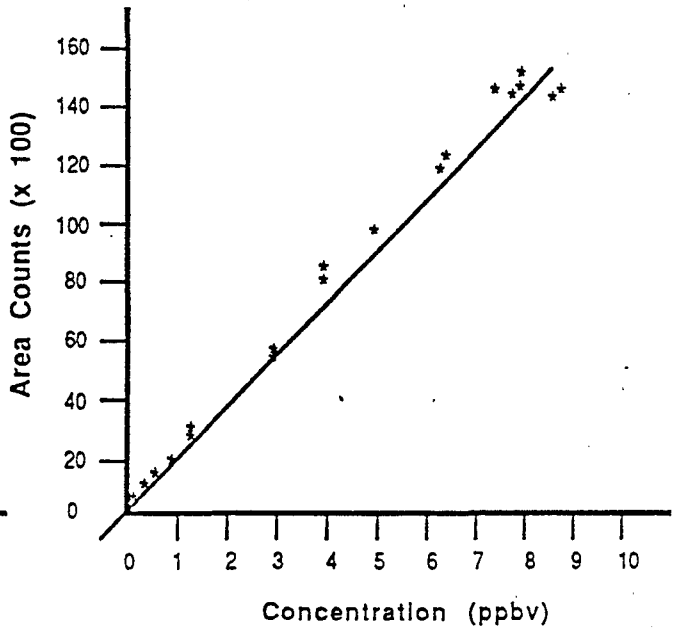


FIGURE 18(d). LINEAR RESPONSE OF CHLOROFORM ON THE ECD

FIGURE 18. RESPONSE OF ECD TO VARIOUS VOCs

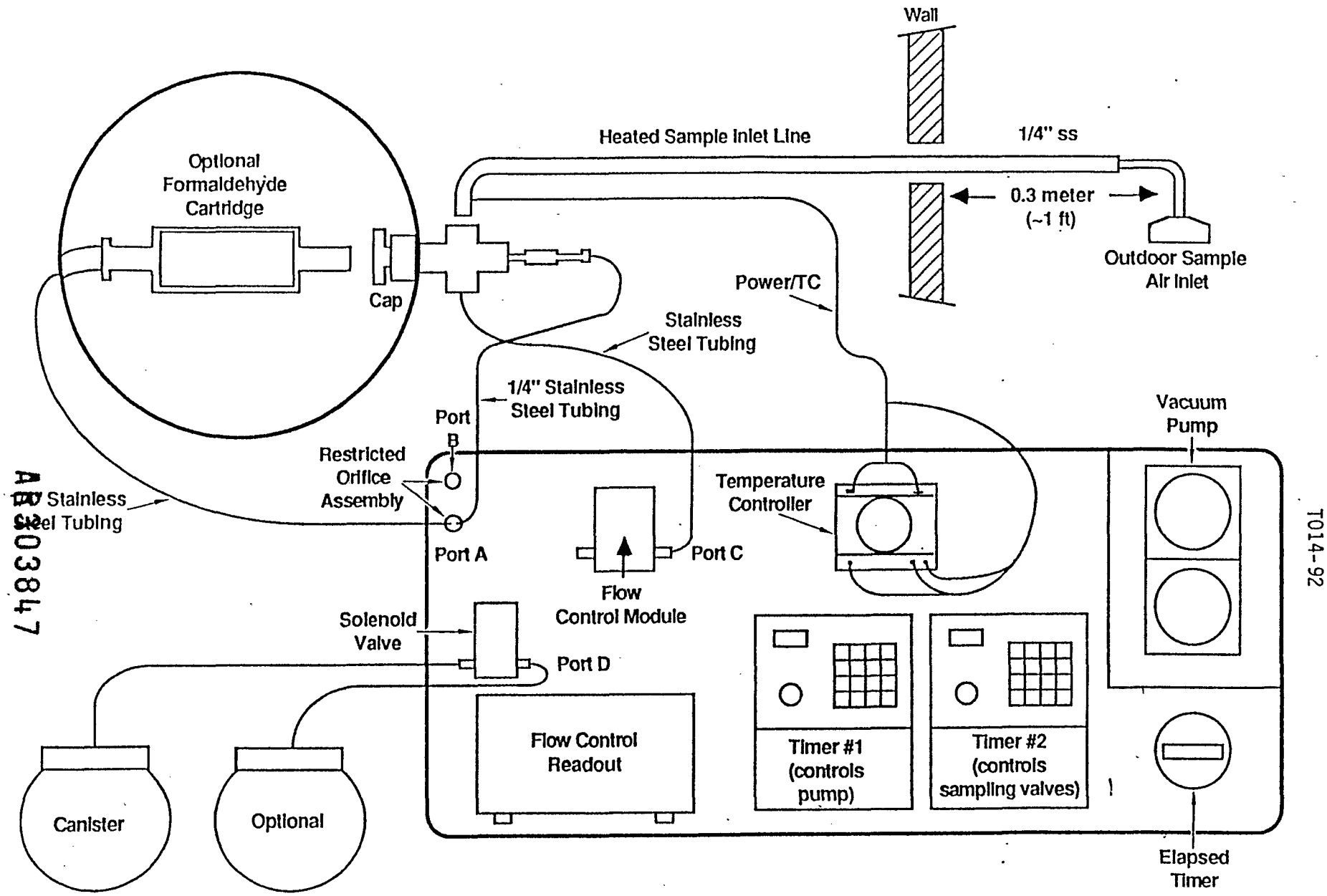


FIGURE 19. U.S. ENVIRONMENTAL PROTECTION AGENCY UATP, SCHEMATIC OF SAMPLE INLET CONNECTIONS SAMPLER

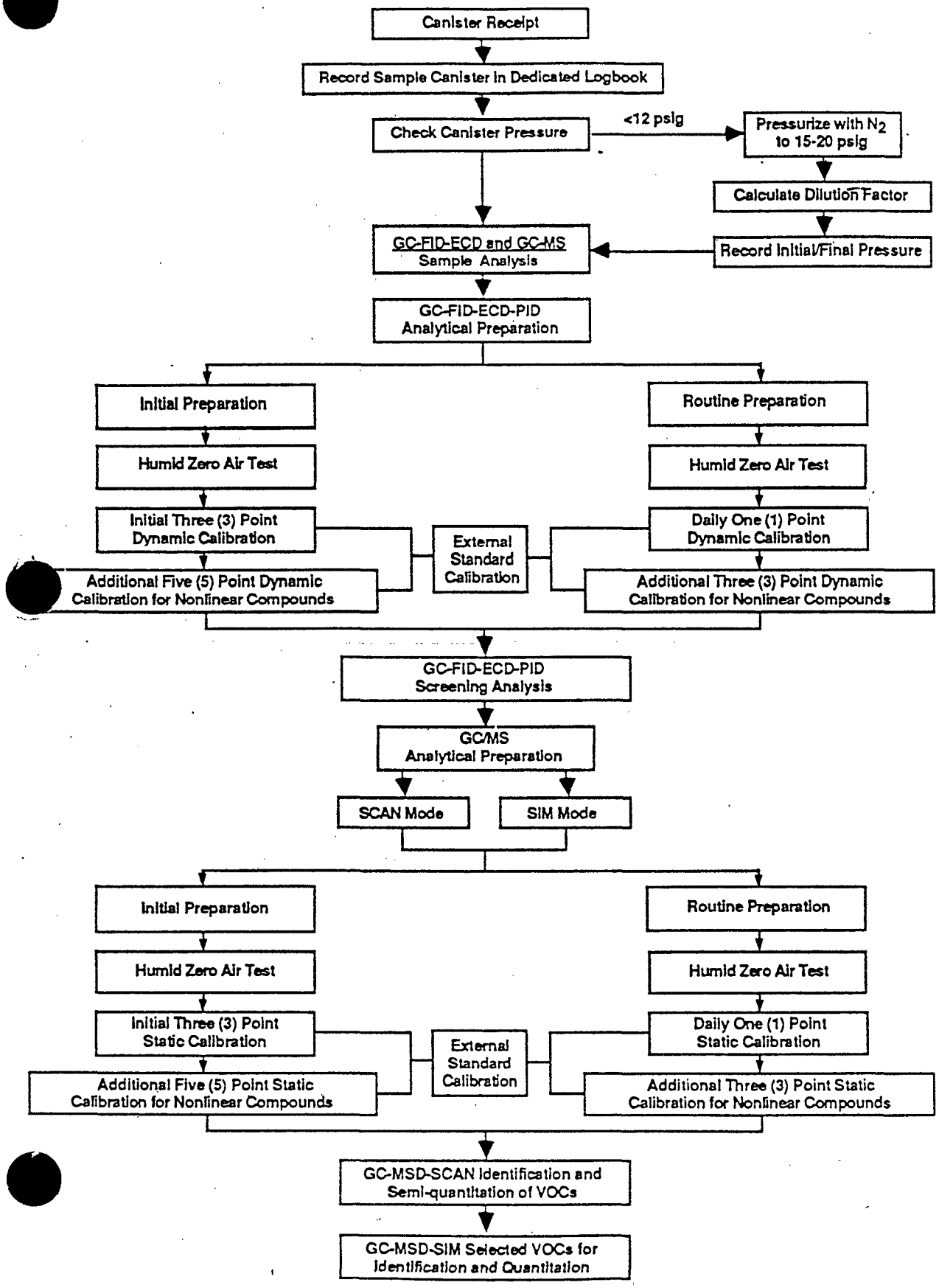
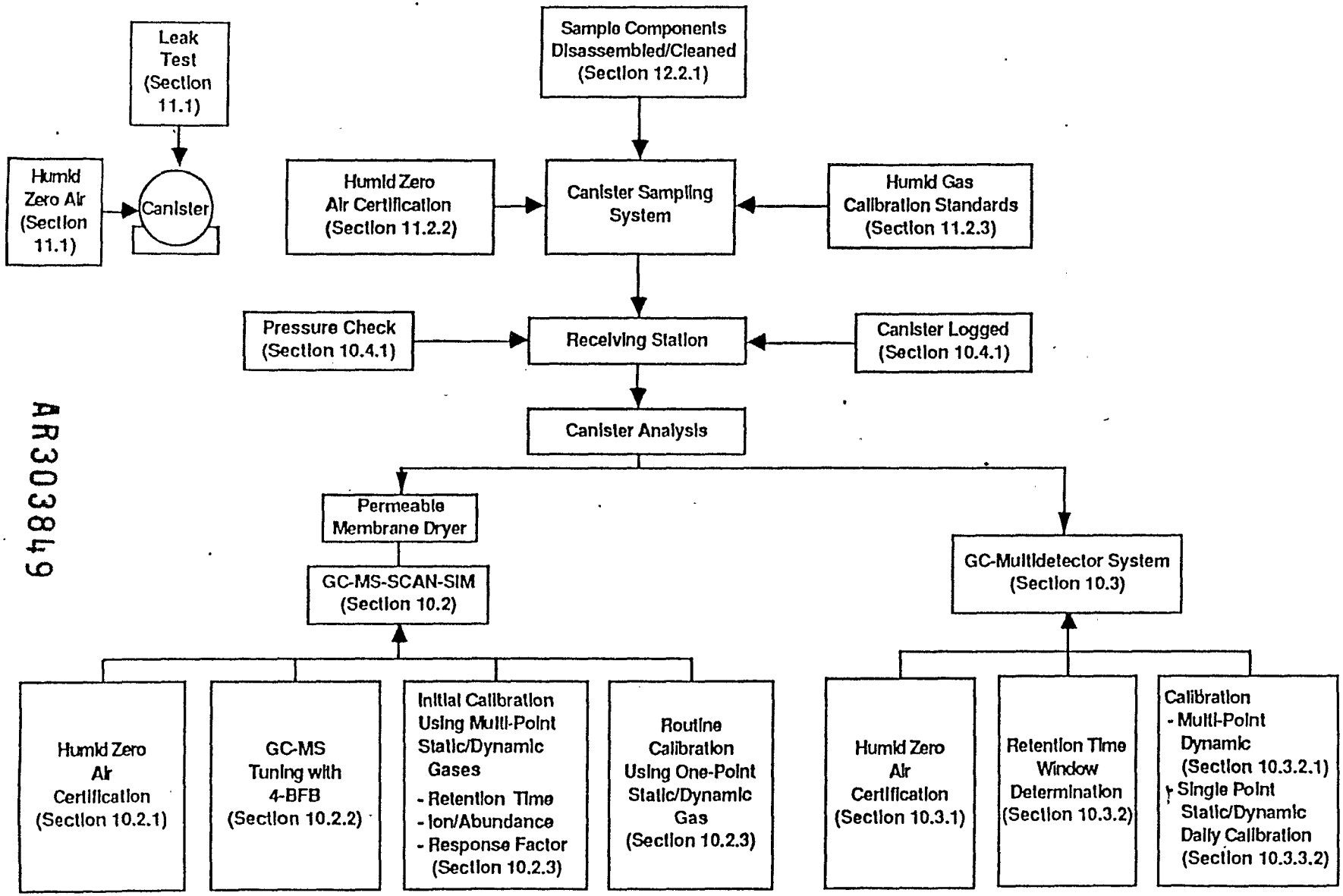


FIGURE 20. FLOWCHART OF ANALYTICAL SYSTEMS PREPARATION.



AR303849

T014-94

FIGURE 21. SYSTEM QUALITY ASSURANCE / QUALITY CONTROL (QA/QC) ACTIVITIES ASSOCIATED WITH VARIOUS ANALYTICAL SYSTEMS

APPENDIX A

AVAILABILITY OF AUDIT CYLINDERS FROM UNITED STATES ENVIRONMENTAL PROTECTION AGENCY USEPA PROGRAMS/ REGIONAL OFFICES, STATE AND LOCAL AGENCIES AND THEIR CONTRACTORS

1. Availability of Audit Cylinders

   1.1 The USEPA has available, at no charge, cylinder gas standards of hazardous organic compounds at the ppb level that may be used to audit the performance of ambient air source measurement systems.

   1.2 Each audit cylinder contains 5 to 18 hazardous organic compounds in a balance of N_2 gas. Audit cylinders are available in several concentration ranges. The concentration of each organic compound in the audit cylinder is within the range illustrated in Table A-1.

2. Audit Cylinder Certification

   2.1 All audit cylinders are periodically analyzed to assure that cylinder concentrations have remained stable.

   2.2 All stability analyses include quality control analyses of ppb hazardous organic gas standards prepared by the National Bureau of Standards for USEPA.

3. Audit Cylinder Acquisition

   3.1 USEPA program/regional offices, state/local agencies, and their contractors may obtain audit cylinders (and an audit gas delivery system, if applicable) for performance audits during:

   - o RCRA Hazardous Waste Trial Burns For PHOC's; and
   - o Ambient Air Measurement of Toxic Organics.

   3.2 The audit cylinders may be acquired by contacting:

Robert L. Lampe
U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory
Quality Assurance Division
MD-77B
Research Triangle Park, NC 27711
919-541-4531

TABLE A-1. AVAILABLE USEPA PERFORMANCE AUDIT CYLINDERS

Group I Compounds	Group II Compounds	Group III Compounds
Carbon tetrachloride	Trichloroethylene	Pyridine (Pyridine in Group III cylinders but certified analysis not available)
Chloroform	1,2-dichloroethane	Vinylidene chloride
Perchloroethylene	1,2-dibromoethane	1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113)
Vinyl chloride	Acetonitrile	1,2-dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
Benzene	Trichlorofluoromethane (Freon-11)	Acetone
	Dichlorodifluoromethane (Freon-12)	1-4 Dioxane
	Bromomethane	Toluene
	Methyl ethyl ketone	Chlorobenzene
	1,1,1-trichloroethane	

Group I Ranges	Group II Ranges	Group III Ranges
7 to 90 ppb	7 to 90 ppb	7 to 90 ppb
90 to 430 ppb	90 to 430 ppb	90 to 430 ppb
430 to 10,000 ppb		

Group IV	Group V	
Acrylonitrile	Carbon tetrachloride	Methylene chloride
1,3-butadiene	Chloroform	Trichlorofluoromethane (Freon-11)
Ethylene oxide	Perchloroethylene	Bromomethane
Methylene chloride	Vinyl chloride	Toluene
Propylene oxide	Benzene	Chlorobenzene
o-xylene	Trichloroethylene	1,3-Butadiene
	1,2-dichloroethane	o-xylene
	1,2-dibromoethane	Ethyl benzene
	1,1,1-trichloroehtane	1,2-dichloropropane

Group IV Ranges	Group V Ranges
7 to 90 ppb	1 to 40 ppb
430 to 10,000 ppb	

APPENDIX B

OPERATING PROCEDURES FOR A PORTABLE GAS CHROMATOGRAPH EQUIPPED WITH A PHOTOIONIZATION DETECTOR

1. Scope

This procedure is intended to screen ambient air environments for volatile organic compounds. Screening is accomplished by collection of VOC samples within an area and analysis on site using a portable gas chromatograph/integrator (Photovac Models 10S10, 10S50, or equivalent). This procedure is not intended to yield quantitative or definite qualitative information regarding the substances detected. Rather, it provides a chromatographic "profile" of the occurrence and intensity of unknown volatile compounds which assists in placement of fixed-site samplers.

2. Applicable Documents

2.1 ASTM Standards

E260 - Recommended Practice for General Gas Chromatography Procedures

E355 - Practice for Gas Chromatography Terms and Relationships

2.2 Other Documents

Portable Instruments User's Manual for Monitoring VOC Sources, EPA-34011-86-015, U.S. Environmental Protection Agency, Washington, DC, June, 1986.

3. Summary of Method

3.1 An air sample is extracted directly from ambient air and analyzed on site by a portable GC.

3.2 Analysis is accomplished by drawing an accurate volume of ambient air through a sampling port and into a concentrator, then the sample air is transported by carrier gas onto a packed column and into a PID, resulting in response peak(s). Retention times are compared with those in a standard chromatogram to predict the probable identity of the sample components.

Significance

4.1 VOCs are emitted into the atmosphere from a variety of sources including petroleum refineries, synthetic organic chemical plants,

natural gas processing plants, and automobile exhaust. Many of these VOC emissions are acutely toxic; therefore, their determination in ambient air is necessary to assess human health impacts.

4.2 Conventional methods for VOC determination use solid sorbent and canister sampling techniques.

4.3 Collection of ambient air samples in canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples, if necessary; (4) unattended sample collection; (5) analysis of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of samples by several analytical systems.

4.4 The use of portable GC equipped with multidetectors has assisted air toxics programs by using the portable GC as a "screening tool" to determine "hot spots," potential interferences, and semi-quantitation of VOCs/SVOCs, prior to locating more traditional fixed-site samplers.

5. Definitions

Definitions used in this document and in any user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Methods D1356 and E355. Abbreviations and symbols pertinent to this method are defined at point of use.

6. Interferences

6.1 The most significant interferences result from extreme differences in limits of detection (LOD) among the target VOCs (Table B-1). Limitations in resolution associated with ambient temperature, chromatography and the relatively large number of chemicals result in coelution of many of the target components. Coelution of compounds with significantly different PID sensitivities will mask compounds with more modest sensitivities. This will be most dramatic in interferences from benzene and toluene.

6.2 A typical chromatogram and peak assignments of a standard mixture of target VOCs (under the prescribed analytical conditions of this method) are illustrated in Figure B-1. Samples which contain a highly complex mixture of components and/or interfering levels of benzene and toluene are analyzed on a second, longer chromatographic column. The same liquid phase in the primary column is contained in the alternate column but at a higher percent loading.

6.3 Recent designs in commercially available GCs (Table B-2) have pre-concentrator capabilities for sampling lower concentrations of VOCs, pre-column detection with back-flush capability for shorter analytical time, constant column temperature for method precision and accuracy and multidetector (PID, ECD, and FID) capability for versatility. Many of these newer features address the weaknesses and interferences mentioned above.

7. Apparatus

7.1 Gas chromatograph. A GC (Photovac Inc., 739 B Parks Ave, Huntington, NY, 11743, Model 10S10 or 10S50, or equivalent) used for surveying ambient air environments (which could employ a multidetector) for sensing numerous VOCs compounds eluting from a packed column at ambient temperatures. This particular portable GC procedure is written employing the photoionization detector as its major sensing device, as part of the Photovac Model 10S10 portable GC survey tool. Chromatograms are developed on a column of 3% SP-2100 on 100/120 Supelcoport (0.66 m x 3.2 mm I.D.) with a flow of 30 cm^3/min air.

7.2 GC accessories. In addition to the basic gas chromatograph, several other pieces of equipment are required to execute the survey sampling. Those include gas-tight syringes for standard injection, alternate carrier gas supplies, high pressure connections for filling the internal carrier gas reservoir, and if the Model 10S10 is used, a recording integrator (Hewlett Packard, Avondale, PA, Model 3390A, or equivalent).

8. Reagents and Materials

8.1 Carrier gas. "Zero" air [<0.1 ppm total hydrocarbon (THC)] is used as the carrier gas. This gas is conveniently contained in 0.84 m^3 (30 ft^3) aluminum cylinders. Carrier gas of poorer quality

may result in spurious peaks in sample chromatograms. A Brooks, Type 1355-00F1AAA rotameter (or equivalent) with an R-215-AAA tube and glass float is used to set column flow.

8.2 System performance mixture. A mixture of three target compounds (e.g., benzene, trichloroethylene, and styrene) in nitrogen is used for monitoring instrument performance. The approximate concentration for each of the compounds in this mixture is 10 parts per billion (ppb). This mixture is manufactured in small, disposable gas cylinders [at 275 kPa (40 psi)] from Scott Specialty Gases, or equivalent.

8.3 Reagent grade nitrogen gas. A small disposable cylinder of high purity nitrogen gas is used for blank injections.

8.4 Sampling syringes. Gas-tight syringes, without attached shut-off valves (Hamilton Model 1002LT, or equivalent) are used to introduce accurate sample volumes into the high pressure injectors on the portable gas chromatograph. Gas syringes with shut-off valves are not recommended because of memory problems associated with the valves. For samples suspected of containing high concentrations of volatile compounds, disposable glass syringes (e.g, Glaspak, or equivalent) with stainless steel/Teflon® hub needles are used.

8.5 High pressure filler. An adapter (Photovac SA101, or equivalent) for filling the internal carrier gas reservoir on the portable GC is used to deliver "zero" air.

9. Procedure

9.1 Instrument Setup

9.1.1 The portable gas chromatograph must be prepared prior to use in the ambient survey sampling. The pre-sampling activities consist of filling the internal carrier gas cylinder, charging the internal power supply, adjusting individual column carrier gas flows, and stabilizing the photoionization detector.

9.1.2 The internal reservoir is filled with "zero" air. The internal 12V, 6AH lead/acid battery can be recharged to provide up to eight hours of operation. A battery

which is discharged will automatically cause the power to the instrument to be shut down and will require an overnight charge. During AC operation, the batteries will automatically be trickle-charged or in a standby mode.

9.1.3 The portable GC should be operated (using the internal battery power supply) at least forty minutes prior to collection of the first sample to insure that the photoionzation detector has stabilized. Upon arriving at the area to be sampled, the unit should be connected to AC power, if available.

9.2 Sample Collection

9.2.1 After the portable gas chromatograph is located and connected to 110V AC, the carrier gas flows must be adjusted. Flows to the 1.22 meter, 5% SE-30 and 0.66 meter, 3% SP2100 columns are adjusted with needle valves. Flows of 60 cm^3/min (5% SE-30) and 30 cm^3/min (3% SP2100) are adjusted by means of a calibrated rotameter. Switching between the two columns is accomplished by turning the valve located beneath the electronic module. During long periods of inactivity, the flows to both columns should be reduced to conserve pressure in the internal carrier gas supply. The baseline on the recorder/integrator is set to 20% full scale.

9.2.2 Prior to analysis of actual samples, an injection of the performance evaluation mixture must be made to verify chromatographic and detector performance. This is accomplished by withdrawing 1.0 mL samples of this mixture from the calibration cylinder and injecting it onto the 3% SP2100 column. The next sample analyzed should be a blank, consisting of reagent grade nitrogen.

9.2.3 Ambient air samples are injected onto the 3% SP2100 column. The chromatogram is developed for 15 minutes. Samples which produce particularly complex chromatograms,

especially for early eluting components, are reinjected on the 5% SE-30 column. [Note: In no instance should a syringe which has been used for the injection of the calibrant/system performance mixture be used for the acquisition and collection of samples, or vice versa.]

9.2.4 Samples have generally been collected from the ambient air at sites which are near suspected sources of VOCs and SVOCs and compared with those which are not. Typically, selection of sample locations is based on the presence of chemical odors. Samples collected in areas without detectable odors have not shown significant PID responses. Therefore, sampling efforts should be initially concentrated on "suspect" environments (i.e., those which have appreciable odors). The objective of the sampling is to locate sources of the target compounds. Ultimately, samples should be collected throughout the entire location, but with particular attention given to areas of high or frequent occupation.

9.3 Sample Analysis

9.3.1 Qualitative analysis. Positive identification of sample components is not the objective of this "screening" procedure. Visual comparison of retention times to those in a standard chromatogram (Figure B-1) are used only to predict the probable sample component types.

9.3.2 Estimation of levels. As with qualitative analysis, estimates of component concentrations are extremely tentative and are based on instrument responses to the calibrant species (e.g., benzene, trichloroethylene, styrene), the proposed component identification, and the difference in response between sample component and calibrant. For purposes of locating pollutant emission sources, roughly estimated concentrations and suspected compound types are considered sufficient.

10. Performance Criteria and Quality Assurance

Required quality assurance measures and guidance concerning performance criteria that should be achieved within each laboratory are summarized and provided in the following section.

10.1 Standard Operating Procedures

10.1.1 SOPs should be generated by the users to describe and document the following activities in their laboratory: (1) assembly, calibration, leak check, and operation of the specific portable GC sampling system and equipment used; (2) preparation, storage, shipment, and handling of the portable GC sampler; (3) purchase, certification, and transport of standard reference materials; and (4) all aspects of data recording and processing, including lists of computer hardware and software used.

10.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the personnel conducting the survey work.

10.2 Quality Assurance Program

10.2.1 Reagent and materials control. The carrier gas employed with the portable GC is "zero air" containing less than 0.1 ppm VOCs. System performance mixtures are certified standard mixtures purchased from Scott Specialty Gases, or equivalent.

10.2.2 Sampling protocol and chain of custody. Sampling protocol sheets must be completed for each sample. Specifics of the sample with regard to sampling location, sample volume, analysis conditions, and supporting calibration and visual inspection information are detailed by these documents. An example form is exhibited in Table B-3.

10.2.3 Blanks, Duplicates, and System Performance Samples

10.2.3.1 Blanks and Duplicates. Ten percent of all injections made to the portable GC are blanks,

where the blank is reagent grade nitrogen gas. This is the second injection in each sampling location. An additional 10% of all injections made are duplicate injections. This will enhance the probability that the chromatogram of a sample reflects only the composition of that sample and not any previous injection. Blank injections showing a significant amount of contaminants will be cause for remedial action.

10.2.3.2 System Performance Mixture. An injection of the system performance mixture will be made at the beginning of a visit to a particular sampling location (i.e., the first injection). The range of acceptable chromatographic system performance criteria and detector response is shown in Table B-4. These criteria are selected with regard to the intended application of this protocol and the limited availability of standard mixtures in this area. Corrective action should be taken with the column or PID before sample injections are made if the performance is deemed out-of-range. Under this regimen of blanks and system performance samples, approximately eight samples can be collected and analyzed in a three hour visit to each sampling location.

10.3 Method Precision and Accuracy

The purpose of the analytical approach outlined in this method is to provide presumptive information regarding the presence of selected VOCs and SVOCs emissions. In this context, precision and accuracy are to be determined. However, quality assurance criteria are described in Section 10.2 which insure the samples collected represent the ambient environment.

10.4 Range and Limits of Detection

The range and limits of detection of this method are highly compound dependent due to large differences in response of the portable GCs photoionization detectors to the various

target compounds. Aromatic compounds and olefinic halogenated compounds will be detected at lower levels than the halomethanes or aliphatic hydrocarbons. The concentration range of application of this method is approximately two orders of magnitude.

TABLE B-1

ESTIMATED LIMITS OF DETECTION (LOD) FOR SELECTED VOCs
BASED ON 1 uL SAMPLE VOLUME

Compound	LOD (ng)	LOD (ppb)
Chloroform[a]	2	450
1,1,1-Trichloroethane[a]	2	450
Carbon tetrachloride[a]	2	450
Benzene	.006	2
1,2-Dichloroethane[b]	.05	14
Trichloroethylene[b]	.05	14
Tetrachloroethylene[b]	.05	14
1,2-Dibromoethane	.02	2
p-Xylene[c]	.02	4
m-Xylene[c]	.02	4
o-Xylene[d]	.01	3
Styrene[d]	.01	3

[a]Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.
[b]1,2-Dichloroethane, Tricholroethylene, and Tetrachloroethylene coelute on 0.66 m 3% SP2100.
[c]p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.
[d]Styrene and o-Xylene coelute on 0.66 m 3% SP2100.

TABLE B-2

COMMERCIALLY AVAILABLE PORTABLE VOC DETECTION INSTRUMENTS

Monitor	Detection principle	Range, ppm	Sensitivity	Response time, s	Accessories	Calibration Techniques	Weaknesses	Service Rate	Lack of Response	Cost, $	Samp Rate L/m
550,551 555,580 (AID, Inc.	PID, FID	0-200, 0-2000, 0-10,000	0.1 ppm at 0-200 ppm	<5		o Bag Sampling	o Umbilical cord too short o Digital readout hard to read o Flame out frequently	8 hrs		4,300	1.5
OVA 108, 128 Century Systems, Inc. (Foxboro)	FID	0-10, 0-100, 0-1000, 0-10,000, 0-100,000	0.2 ppm (Model 128) 0.5 ppm (Model 108)	2 2	o Thermal Desorbers available o Optional GC available	o Hand Space o Direct Injection o Bag Samp.	o Battery failure o Sample line kinks o Compounds containing O_2/N give low response o Neg. resp. to CO/CO_2	8 hrs		6,300	
[illegible]rs-Inc)	PID	1 1-20 1-200 1-2000	0.1 ppm Low molecular weights aromatics	<5	o Three lamps available o 9.5 (aromatics) o 10.2 (2-4 compounds) o 11.7 (halocarbons)	o External Gas Cyl. o Bag Samp.	o Three lamps - may miss something	10 hrs	o Cl hydrocarbons o CH_4	4,955	0.5
TLV Sniffer (Bacharach)	Catalytic combustion	0-500 0-5000 0-50,000	2.0 ppm	5		o Bag Samp. o Head Space				900	
Ecolyzer 400 (Energetics Science)	Catalytic combustion	0-100% LFL	1% LFL	15		o Bag Samp.	o Changes in gas temp/ humidity affects response				
Miran 1A (Foxboro)	IR	ppm to %	1 ppm	1,4,10 and 40						9,500	
Miran 1B (Foxboro)	IR	ppm to %								12,500	
Scentor (Sentex)	GC/EC, Argon Ionization PID		0.01 ppb Cl organics	2	Preconcentrator Thermal Desoprtion GC Columns Auto Cal. from Integral Gas Cylinder	o Internal gas cyl. o Preconcentrator o GC Column				12,950	
Photovac Standard Automatic Computer Auto Comp. Communication	PID (UV Light)	0	0.1 ppb Benzene with signal-to-noise ratio 4:1, Good for aromatics	2	o Dual Column o Manual/Auto Injection o Column Cond. o Pre-flush o Auto Dial Modem o Programmable		o Column operates at ambient temp. o STD in lab, then to field at diff. temp o Can't inject liquid samp. o Light fractions interfere		o H_2O o O_2	6,995 8,995 10,500 10,955 12,955	
Photovac Tip	PID	0-2000 ppm	0.05 ppm Benzene	3							

TABLE B-3

PORTABLE GAS CHROMATOGRAPH
SAMPLING DATA SHEET

DATE: _____ LOCATION: _____ TIME: _____

CHROMATOGRAPHIC CONDITIONS: _____

COLUMN 1: COLUMN TYPE:

I.D. (mm):_____ LENGTH (mm):_____ FLOW (mL/min):_____

COLUMN 2: COLUMN TYPE:

I.D. (mm):_____ LENGTH (mm):_____ FLOW (mL/min):_____

INJ. NO.	INJ. VOL.	COLUMN NO.	SETTING	LOCATION

SITE PLAN (indicate sampling locations):

_____ DATE

_____ SIGNATURE

TABLE B-4

SYSTEM PERFORMANCE CRITERIA FOR PORTABLE GC[a]

Criteria	Test Compound	Acceptable Range	Suggested Corrective Action
PID Response	Trichloroethylene	$\geq 10^8$ uV-sec/ng	Re-tune or replace lamp
Elution Time	Styrene	2.65 $\pm$ 0.15 min	Inspect for leaks, adjust carrier flow
Resolution[b]	Benzene/Trichloroethylene	$\geq$ 1.4	Replace column

[a]Based on analysis of a vapor mixture of benzene, styrene, and trichloroethylene.

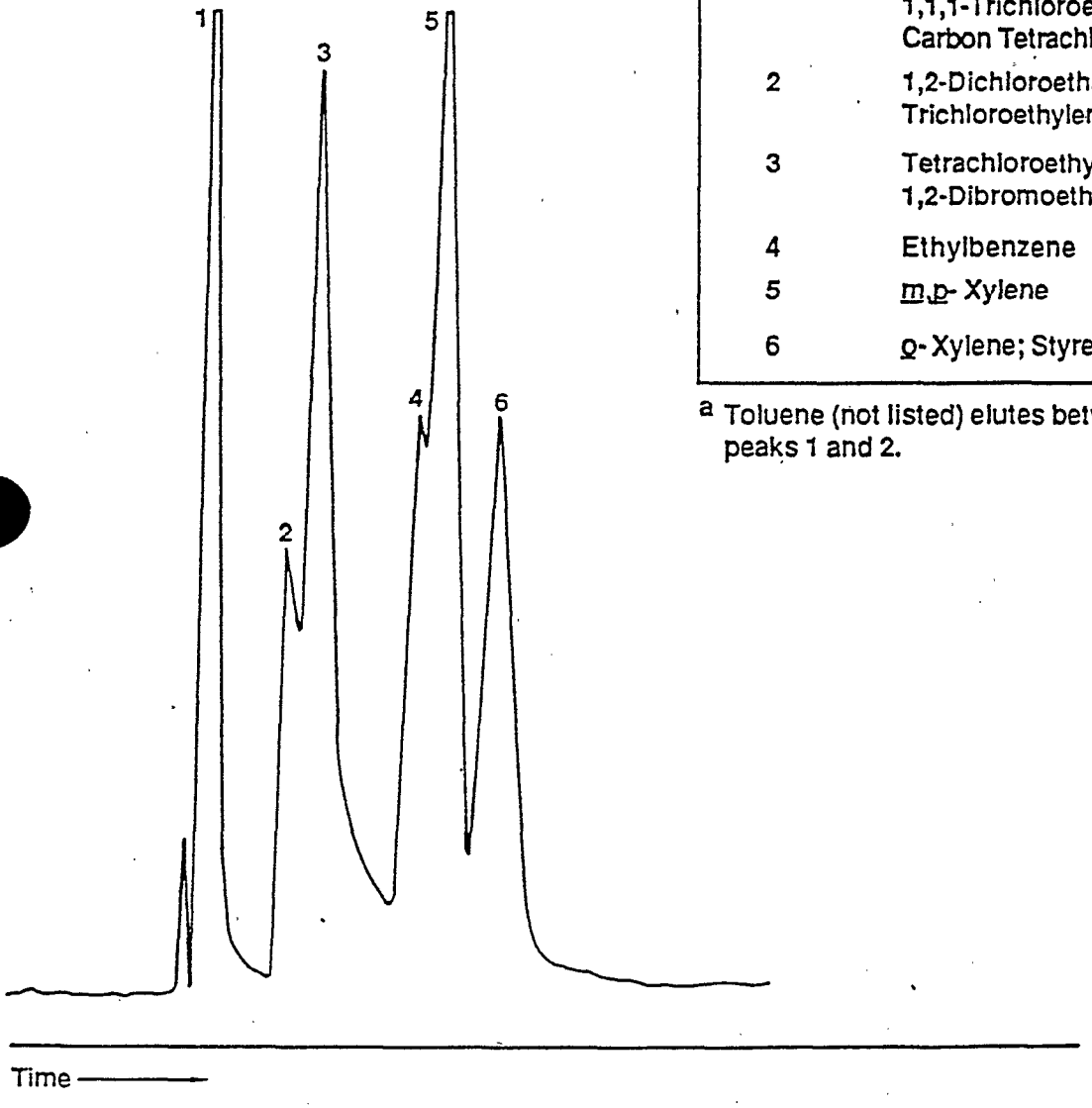
[b]Define by: $R + = 2d/(W_1+W_2)$; where d = distance between the peaks and W = peak width at base.

TABLE B-5

ESTIMATED LIMITS OF DETECTION (LOD) FOR SELECTED VOCs

Compound	LOD (ng)	LOD (ppb)
Chloroform[a]	2	450
1,1,1-Trichloroethane[a]	2	450
Carbon tetrachloride[a]	2	450
Benzene	.006	2
1,2-Dichloroethane[b]	.05	14
Trichloroethylene[b]	.05	14
Tetrachloroethylene[b]	.05	14
1,2-Dibromoethane	.02	2
p-Xylene[c]	.02	4
m-Xylene[c]	.02	4
o-Xylene[d]	.01	3
Styrene[d]	.01	3

[a]Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.
[b]1,2-Dichloroethane, Trichloroethylene, and Tetrachloroethylene coelute on 0.66 m 3% SP2100.
[c]p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.
[d]Styrene and o-Xylene coelute on 0.66 m 3% SP2100.



Peak Assignments For Standard Mixture

Peak No.	Compound(s) [a]
1	Benzene; Chloroform; 1,1,1-Trichloroethane; Carbon Tetrachloride
2	1,2-Dichloroethane; Trichloroethylene
3	Tetrachloroethylene; 1,2-Dibromoethane
4	Ethylbenzene
5	m,p- Xylene
6	o- Xylene; Styrene

[a] Toluene (not listed) elutes between peaks 1 and 2.

FIGURE B-1. TYPICAL CHROMATOGRAM OF VOCs DETERMINED BY A PORTABLE GC

APPENDIX C

INSTALLATION AND OPERATION PROCEDURES FOR U.S. ENVIRONMENTAL PROTECTION AGENCY'S URBAN AIR TOXIC POLLUTANT PROGRAM SAMPLER

1. Scope

1.1 The subatmospheric sampling system described in this method has been modified and redesigned specifically for use in USEPA's Urban Air Toxic Pollutant Program (UATP), a joint project of USEPA's Office of Air Quality Planning and Standards, the Environmental Monitoring Systems Laboratory, and the participating state air pollution control agencies. The purpose of UATP is to provide analytical support to the states in their assessment of potential health risks from certain toxic organic compounds that may be present in urban atmospheres. The sampler is described in the paper, "Automatic Sampler for Collection of 24-Hour Integrated Whole-Air Samples for Organic Analysis," to be presented at the 1988 Annual Meeting of APCA, Dallas, Texas, June, 1988 (Paper No. 88-150.3).

1.2 The sampler is based on the collection of whole air samples in 6-liter, SUMMA® passivated stainless steel canisters. The sampler features electronic timer for ease, accuracy and flexibility of sample period programming, an independently setable presample warm-up and ambient air purge period, protection from loss of sample due to power interruptions, and a self-contained configuration housed in an all-metal portable case, as illustrated in Figure C-1.

1.3 The design of the sampler is pumpless, using an evacuated canister to draw the ambient sample air into itself at a fixed flow rate (3-5 cm^3/min) controlled by an electronic mass flow controller. Because of the relatively low sample flow rates necessary for the integration periods, auxiliary flushing of the sample inlet line is provided by a small, general-purpose vacuum pump (not in contact with the sample air stream). Further, experience has shown that inlet lines and surfaces sometimes build up or accumulate substantial concentrations of organic materials under stagnant (zero flow rate) conditions. Therefore such lines and surfaces need to be purged and equilibrated to the sample air for some time prior to the beginning of the actual sample collection period. For this reason, the sampler includes dual timers, one of which is set to start the pump several hours prior to the specified start of the sample period to purge the inlet lines and

surfaces. As illustrated in Figure C-1, sample air drawn into the canister passes through only four components: the heated inlet line, a 2-micron particulate filter, the electron flow controller, and the latching solenoid valve.

2. Summary of Method

2.1 In operation, timer 1 is set to start the pump about 6 hours before the scheduled sample period. The pump draws sample air in through the sample inlet and particulate filter to purge and equilibrate these components, at a flow rate limited by the capillary to approximately 100 cm^3/min. Timer 1 also energizes the heated inlet line to allow it to come up to its controlled temperature of 65 to 70 degrees C, and turns on the flow controller to allow it to stabilize. The pump draws additional sample air through the flow controller by way of the normally open port of the 3-way solenoid valve. This flow purges the flow controller and allows it to achieve a stable controlled flow at the specified sample flow rate prior to the sample period.

2.2 At the scheduled start of the sample period, timer 2 is set to activate both solenoid valves. When activated, the 3-way solenoid valve closes its normally open port to stop the flow controller purge flow and opens its normally closed port to start flow through the aldehyde sample cartridges. Simultaneously, the latching solenoid valve opens to start sample flow into the canister.

2.3 At the end of the sample period, timer 2 closes the latching solenoid valve to stop the sample flow and seal the sample in the canister and also de-energizes the pump, flow controller, 3-way solenoid, and heated inlet line. During operation, the pump and sampler are located external to the sampler. The 2.4 meter (8 foot) heated inlet line is installed through the outside wall, with most of its length outside and terminated externally with an inverted glass funnel to exclude precipitation. The indoor end is terminated in a stainless steel cross fitting to provide connections for the canister sample and the two optional formaldehyde cartridge samples.

3. Sampler Installation

3.1 The sampler must be operated indoors with the temperature between 20-32°C (68 to 90°F). The sampler case should be located conveniently

on a table, shelf, or other flat surface. Access to a source of 115 vac line power (500 watts min) is also required. The pump is removed from the sampler case and located remotely from the sampler (connected with a 1/4 inch O.D. extension tubing and a suitable electrical extension cord).

3.2 Electrical Connections (Figure C-1)

3.2.1 The sampler cover is removed. The sampler is not plugged into the 115 vac power until all other electrical connections are completed.

3.2.2 The pump is plugged into its power connector (if not already connected) and the battery connectors are snapped onto the battery packs on the covers of both timers.

3.2.3 The sampler power plug is inserted into a 115 volts ac line grounded receptacle. The sampler must be grounded for operator safety. The electrical wires are routed and tied so they remain out of the way.

3.3 Pneumatic Connections

3.3.1 The length of 1/16 inch O.D. stainless steel tubing is connected from port A of the sampler (on the right side of the flow controller module) to the air inlet line.

3.3.2 The pump is connected to the sampler with 1/4 inch O.D. plastic tubing. This tubing may be up to 7 meters (20 feet) long. A short length of tubing is installed to reduce pump noise. All tubing is conveniently routed and, if necessary, tied in place.

4. Sampler Preparation

4.1 Canister

4.1.1 The sample canister is installed no more than 2 days before the scheduled sampling day.

4.1.2 With timer #1 ON, the flow controller is allowed to warm up for at least 15 minutes, longer if possible.

4.1.3 An evacuated canister is connected to one of the short lengths of 1/8 inch O.D. stainless steel tubing from port B (solenoid valve) of the sampler. The canister valve is left closed. The Swagelok fitting on the canister must not be cross-threaded. The connection is tightened snugly with a wrench.

4.1.4 The end of the other length of stainless steel tubing from port B (solenoid valve) is connected with a Swagelok plug.

4.1.5 If duplicate canisters are to be sampled, the plug is removed from the second 1/8 inch O.D. stainless steel tubing from port B (solenoid valve) and the second canister is connected. The canister valve is left closed.

4.1.6 The ON button of timer #2 is pressed. The flow through the flow controller should be stopped by this action.

4.1.7 The flow controller switch is turned to "READ" and the zero flow reading is obtained. If this reading is not stable, wait until the reading is stabilized.

4.1.8 The flow controller switch is turned to "SET" and the flow setting is adjusted to the algebraic SUM of the most recent entry on Table C-1 and the zero reading obtained in step 4.1.7 (If the zero reading is negative, SUBTRACT the zero reading from the Table C-1 value). Be sure to use the correct Table C-1 flow value for one or two canisters, as appropriate. [Note: If the analytical laboratory determines that the canister sample pressure is too low or too high, a new flow setting or settings will be issued for the sampler. The new flow setting should be recorded in Table C-1 and used until superseded by new settings.]

4.1.9 Timer #2 is turned OFF to again start the flow through the flow controller. With the pump (timer #1) ON and the sampling valve (timer #2) OFF, the flow controller is turned to "READ" and the flow is verified to be the same as the flow setting made in step 4.1.8. If not, the flow setting is rechecked in step 4.1.8 and the flow setting is readjusted if necessary.

4.1.10 The OFF button of timer #1 is pressed to stop the pump.

4.1.11 The canister valve(s) are fully opened.

4.2 Timers

4.2.1 Timer #2 is set to turn ON at the scheduled ON time for the sample period, and OFF at the scheduled OFF time. (See the subsequent section on setting the timers.)
Normal ON time: 12:00 AM on the scheduled sampling day.
Normal OFF time: 11:59 PM on the scheduled sampling day.

(The OFF time is 11:59 PM instead of 12:00 AM so that the day number for the OFF time is the same as the day number for the ON time.) Be sure to set the correct day number.

4.2.2 Timer #1 is set to turn ON six (6) hours before the beginning of the scheduled sample period and OFF at the scheduled OFF time for the sample period (same OFF time as for timer #2). (See the subsequent section on setting the timers.) Normal ON time: 06:00 PM on the day prior to the scheduled sampling day. Normal OFF time: 11:59 PM on the scheduled sampling day. [Note: The timers are wired so that the pump will be on whenever either timer is on. Thus the pump will run if timer #2 is ON even if timer #1 is OFF.]

4.2.3 The elapsed time meter is set to 0.

4.3 Sampler Check

4.3.1 The following must be verified before leaving the sampling site:

(1) Canister(s) is (are) connected properly and the unused connection is capped if only one canister is used.
(2) Canister valve(s) is (are) opened.
(3) Both timers are programmed correctly for the scheduled sample period.
(4) Both timers are set to "AUTO".
(5) Both timers are initially OFF.
(6) Both timers are set to the correct current time of day and day number.
(7) Elasped time meter is set to 0.

4.4 Sampler Recovery (Post Sampling)

4.4.1 The valve on the canister is closed.

4.4.2 The canister is disconnected from the sampler, the sample data sheet is completed, and the canister is prepared for shipment to the analytical laboratory.

4.4.3 If two canisters were sampled, step 2.4.2 is repeated for the other canister.

5. Timer Setting

Since the timers are 7-day timers, the days of the week are numbered from 1 to 7. The assignment of day numbers to days of the week is

indicated on the timer keypad: 1 = Sunday, 2 = Monday, 3 = Tuesday, 4 = Wednesday, 5 = Thursday, 6 = Friday, and 7 = Saturday. This programming is quite simple, but some timers may malfunction or operate erratically if not programmed exactly right. To assure correct operation, the timers should be reset and completely reprogrammed "from scratch" for each sample. The correct current time of day is re-entered to reprogram the timer. Any program in the timer's memory is erased by resetting the timer (pressing the reset button). The timer is set by the following:

(1) pressing the reset button,
(2) entering the correct day number and time of day,
(3) entering the ON and OFF times for the sample period, and
(4) verifying that the ON and OFF time settings are correct.

5.1 Timer Reset

The timer reset button is pressed, which is recessed in a small hole located just above the LED (light emitting diode) indicator light. A small object that will fit through the hole, such as a pencil, match, or pen is used to press the timer. After reset, the timer display should show |1| |10:00|. [Note: The timers may operate erratically when the batteries are discharged, which happens when the sampler is unplugged or without power for several hours. When the sampler is again powered up, several hours may be required to recharge the batteries. To avoid discharging the batteries, the battery pack should be disconnected from the timer when the sampler is unplugged.]

5.2 Date and Time Entry

The selector switch is turned to SET and the number button corresponding to the day number is pressed. (For example, a "2" is pressed for Monday.) The current time of day is entered. (For example, if the time is 9:00 AM, 900 is pressed.) AM or PM is pressed as applicable. (Display should show |2| |'9:00| for 9:00 AM Monday.) [Note: ' indicates AM and indicates PM.] The CLOCK button is pressed. (Display should show |-| |--:--|) If an error is made, |E| |EE:EE| is shown on the display. The CLEAR button is pressed and the above steps are repeated. The selector switch is turned to AUTO or MAN to verify correct time setting.

5.3 ON and OFF Entry

The selector switch is turned to SET. The ON and OFF program is entered in the following order: day, number, time, AM or PM, ON or OFF. (Example: To turn ON at 12:00 AM on day 5 (Thursday); 5, 1200, AM, ON is entered). (Example: To turn OFF at 11:59 PM on day 5 (Thursday), 5, 11:59, PM, OFF is entered.) If the display indicates an error (|E| |EE:EE|), the timer is reset. The selector switch is turned to AUTO.

5.4 ON and OFF Verification

5.4.1 The selector switch is turned to REVIEW. The number of the scheduled sample day is pressed. ON is pressed. The display should show the time of the beginning of the sample period (for example, |5| |'12:00|). [' indicates AM.] ON is pressed again. The display should show |5| |--:--|, indicating no other ON times are programmed.

5.4.2 OFF is pressed. The display should show the time of the end of the sample period, (for example, |5| |, 11:59|). PM is indicated by the "," mark before the time. OFF is pressed again. The display should show |5| |--:--|, indicating no other OFF times are programmed. The selector is switched to AUTO. If anything is incorrect, the timer is reset and reprogrammed.

TABLE C-1

NET FLOW CONTROLLER SETTING

DATE	1 CANISTER	2 CANISTERS

U. S. GOVERNMENT PRINTING OFFICE 1980/640-163/00313

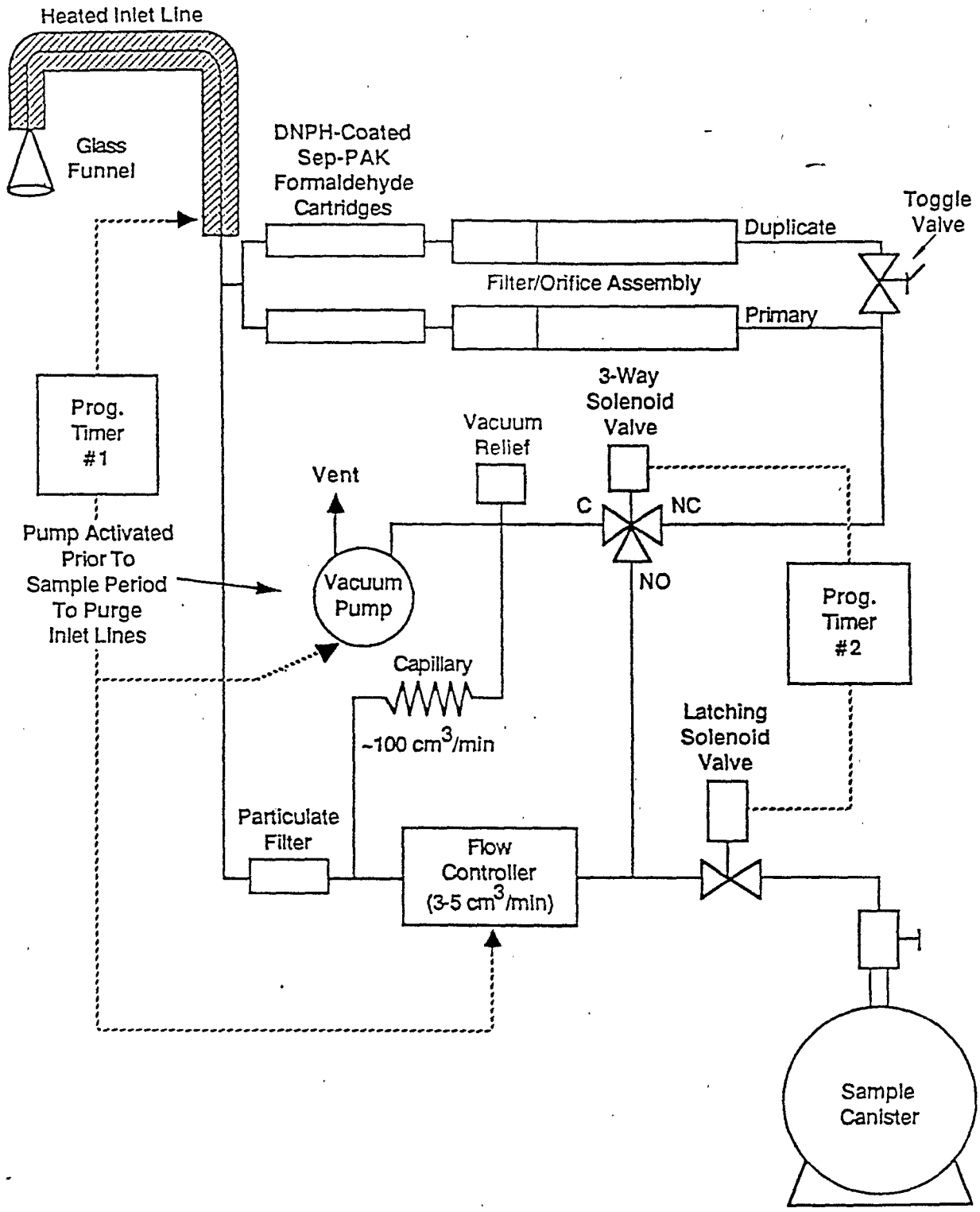


FIGURE C-1. U.S. ENVIRONMENTAL PROTECTION AGENCY UATP SAMPLER SCHEMATIC OF SAMPLE INLET CONNECTIONS

AR303874

APPENDIX C

STANDARD OPERATING PROCEDURES FOR AIR SAMPLING EQUIPMENT

C-1

TEI MODEL 43A PULSED FLUORESCENCE SO_2 ANALYZER AND MODEL 45 PULSED FLUORESCENCE H_2S ANALYZER

TEI MODEL 43A PULSED FLUORESCENCE SO_2 ANALYZER AND MODEL 45 PULSED FLUORESCENCE H_2S ANALYZER

Operation:

1. Connect FEP Teflon, 316 stainless steel, borosilicate glass, or similar tubing, with an outside diameter of one-quarter inch and a minimum inside diameter of one-eighth inch to the sample connector on the back panel of the Model 43A. The length of tubing should be less than 10 feet long.

2. Connect analog recorder to the Model 43A.

3. Toggle the power switch on the front panels of the Model 43A and Model 45 to the "ON" position. The cooling fan, pump, and fluorescent source should now be powered.

4. Swing open the front panel analyzer section door of the Model 43A and verify that the vacuum reading on the pressure gauge is -25.4 cm Hg (minus 10 inches Hg). If necessary, adjust the pressure regulator so that the gauge reads 10.

5. Adjust the temperature knob on the Model 45 to the optimum temperature as supplied by the factory.

6. Allow approximately 20 minutes for the instrument to stabilize before any measurements are made.

7. For SO_2 measurements, set the switch on the Model 45 to the SO_2 setting. Select the 0.1 ppm range setting on the Model 43A. If reading is greater than 0.1 ppm range, increase range setting until reading is below range setting.

8. For H_2S measurements, set the switch on the Model 45 to the SO_x setting. Select the 0.1 ppm range setting on the Model 43A. If reading is greater than 0.1 ppm range, increase range setting until reading is below range setting.

C-2

NATIONAL DRAEGER MODEL 190 DATA-LOGGER GAS MONITOR
(INCLUDING CONVERTER BOX AND ENHANCED GRAPHICS SOFTWARE)

NATIONAL DRAEGER MODEL 190 DATA-LOGGER GAS MONITOR
(Including Converter Box and Enhanced Graphics Software)

Operation

The instrument gives the user the capability to turn logging on and off and to transmit and reset the memory. This is accomplished by fitting a function key to the connector at the top of the instrument. In normal operation, the display continuously scrolls between the concentration, TWA value, and peak value at a fixed interval of time. The instrument also provides audible and visual warning indications at user adjustable limit values. By utilizing the function key on the instrument, the user can perform necessary functions.

Depressing the switch on the red function key once when in the logging mode will disable the logging function and switch from the logging mode to a constant concentration mode. This should be done immediately upon completion of data logging so that unwanted data is not logged.

DEPRESSING THE SWITCH ON THE RED FUNCTION KEY A SECOND TIME WHILE THE INSTRUMENT IS IN CONSTANT CONCENTRATION MODE (ALSO REFERRED TO AS THE 190 MODE), WILL RESET THE LOGGING FUNCTION AND ERASE ALL PREVIOUSLY STORED DATA.

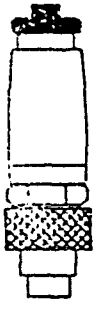
Data stored in dataloggers memory from any logging period must be transmitted to output device (i.e., serial printer or computer) prior to beginning a new logging period, or previously stored logger data will be lost.

Installing the blue reset key on the connector resets the logging function. with the key installed, the display is blank. This can also be used as a power conserving mode (also referred to as a sleep mode) when the logging function is not needed. Removing the blue reset key starts the logging function again. (See Figure 1.)

The unit is continuously operating and ready for use when a battery with sufficient power is connected. Use only alkaline style batteries (9 volt transistor/NEDA 1604A).

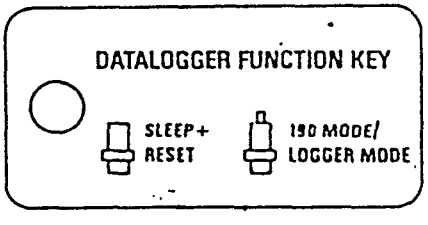
Sufficient batter power is indicated by a periodic flash of the LED visual alarm (approximately every 10 seconds). Under normal operating conditions, the battery will continuously provide power for over 750 hours (one month). When the unit is in alarm condition, more power is drawn from the battery. Excessive alarm conditions will decrease the useful life of the batter. Insufficient battery power is indicated by a short audible tone emitted approximately every 10 seconds. The battery should be replaced at the end of the work shift during which the low battery warning was activated.

Figure 1



FUNCTION KEY
RED

RESET KEY
BLUE



DRAEGER CONVERTER BOX

1. Connect RS232 connector of Converter Box to RS232 port of computer (use suitable extension cable if necessary).

2. Connect AC/DC Adapter to Converter Box and power outlet, making sure Switch 1 is in OFF position (Figure 2).

3. Switch computer on, invoke and configure serial interface driver software. The use of National Draeger Enhanced Graphic Software (P/N 410259) simplifies the configuration and consecutive file handling.

4. Connect Converter Box to Model 190 Datalogger.

5. Switch 1 to ON (green LED on):

   - Transmission starts automatically (red LED flickers).
   - Transmission done (red LED off).

6. When download is done, Switch 1 to OFF:

   - Model 190 Datalogger will go in 190 mode.

7. Disconnect Converter box.

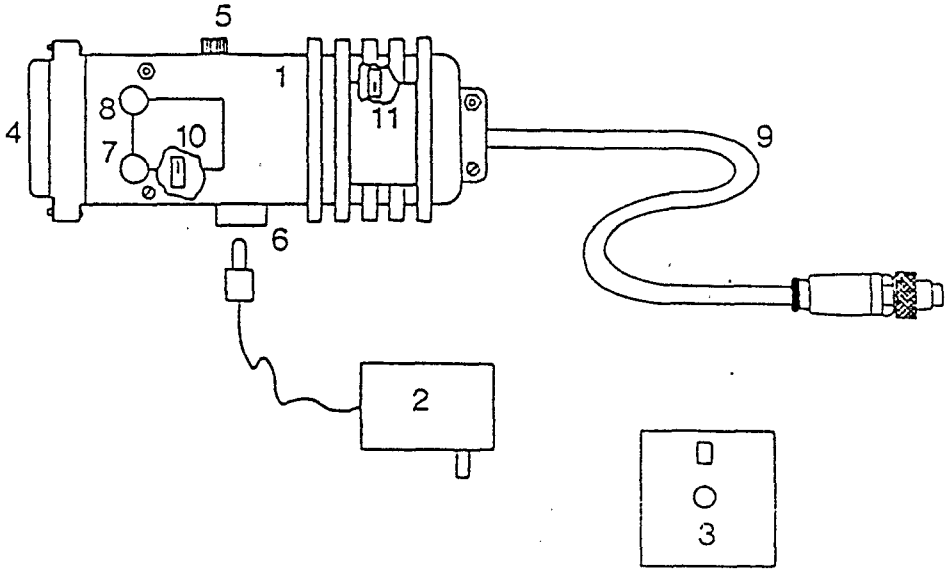
8. Continue with the actual running computer software to get a printout, save data on a disk, or get a graph (only with Enhanced Graphic Software).

9. For additional downloads, repeat Steps 5 & 6.

NOTE: If a download is not successfully done, the Model 190 Datalogger will remain in a "ready to download mode" until a successful download is performed. It is not possible to leave this stage with any function key. This protects the stored data in memory to be deleted by accident. Refer to Troubleshooting Section for solving any problems or disconnect the battery to make a reset (stored data lost).

ORIGINAL (Red)

Figure 2



1	Converter Box
2	9V AC/DC Adaptor
3	Floppy disc - Enhanced Graphic Software
4	RS232 Interface Connector (female)
5	ON/OFF Switch (SW1) for starting transmission
6	AC/DC Supply Connector
7	"Power On" LED (green)
8	"Transmit" LED (red)
9	Interface cable to Model 190 Datalogger
10	Switch 2 (SW2) Selection Data Receive Line
11	Switch 3 (SW3) Connection DTR to ground

Operational Use

Start the computer following the instructions for the PC. Follow the EGS program start-up instructions for the appropriate PC/DRIVE configuration of your system.

When the opening screen with the Draeger logo appears, strike any key to get to the main menu. The different functions may then be performed by typing the related selection number. It is not necessary to press enter after typing the selection number.

List Files (Command 0)

List Files gives a list of existing data files in the directly specified path (See Configuration). Data files are marked with the extension.DAT.

Quit (Command 1)

Quit will exit the program and return to DOS.

Retrieve File (Command 2)

To perform a graph or a printout from an existing data file a file has to be retrieved. Type [2] and enter the filename **without an extension**, or press return and use the cursor arrows to position the cursor on the desired filename (the filename will be highlighted) and press return (Figure 1).

DATALOGGER ANALYZING SYSTEM

Commands:

0 = List Files	1 = Quit
2 = Retrieve_File LOGFILE	3 = Load from Logger
4 = Save File	5 = Display Graph
6 = Print Report	7 = Configuration
8 = Create Database	
Type Command ->	Enter file name ->LOGFILE

Load from Logger (Command 3)

Perform a download from a Datalogger. The program will echo a message to indicate that the program is expecting data at the serial port. Refer to the Converter Box manual to start the download. Receiving data from the Datalogger will be indicted by the message LOADING and a moving cursor on the bottom of the screen. The message ACTIVE indicates the completion of the download and that this file is still active. Default text appears on the screen. The default text information is obtained from the last data file entry. At this point, new text information can be added to the data file. Strike the SPACE bar followed by the ENTER key if no information is desired (Figure 2).

DATALOGGER ANALYZING SYSTEM

Commands:

0 = List Files	1 = Quit
2 = Retrieve File	3 = Load from Logger
4 = Save File	5 = Display Graph
6 = Print Report	7 = Configuration
8 = Create Database	

Type Command ->

Enter Name: ?	0. = undefined
Enter Location: ?	1. = CO
Enter Date: 3/10/90 ?	2. = NO_2
Enter Start Time: 00:00	3. = SO_2
Enter Comment: ?	4. = H_2S
Enter Gas: 0?	
Enter Serial Number: ?	

Name	of user or operator
Location	where measured or headquarter
Date	when logged or downloaded
Start Time	when started logging (military time format)
Comment	regarding this record
Gas	select number from list
Serial Number	of used instrument

Once this information is entered and you advance to the next line, the previous line cannot be edited or manipulated again. Changes may be made only while entering the Start

Time (military time format when logging started), the program will reference the measured concentration to the real time. For additional information about the displayed TWA and STEV, look at the README file included on the program disk.

Save File (Command 4)

When saving a file, enter the filename **without an extension.** The file will be stored in the directory specified in Configuration.

Display Graph (Command 5)

The program creates a graph of the retrieved or downloaded file. The x-axis shows the absolute time. The time format is military (00:00 to 23:59). The y-axis will have a different color for concentration above the horizontal dashed line indicating the limit. The scale-range is automatically set by the highest measured concentration in suitable increments and cannot be changed by the user. The start-up window shows the whole graph and the cursor at the highest sampled concentration. The corresponding time and value of the cursor is displayed in the upper right hand corner of the screen. A dashed horizontal line shows the time weighted average (TWA). The entered information is shown on the bottom of the screen. The following keys perform a special function on the graph:

Function Keys:

F1	zoom in
F2	zoom out
F3	display all
F7	vertical print (landscape)
F8	horizonal print (portrait)
F10	help

Cursor Keys:

←	move cursor left
→	move cursor right
↑	move cursor/window fast left
↓	move cursor/window fast right
PgUp	move half a window left
PgDn	move half a window right
Home	go to the beginning
End	go to the end
Enter	leave graphic mode

Print Report (Command 6)

Perform a printout of the numerical information on a parallel.

Configuration (Command 7)

Define the serial COM port by typing in the number used for the download from the Datalogger. Specify a path pointing to a directory to be used for the storage of data files. It is preset by the factory to C:/EGA/DATA for had drive equipped systems. Refer to Section 3.3 for detailed instructions on installation and configuration for hard drive equipped systems and Sections 3.4 and 3.5 for dual and single floppy disk drive systems.

C-3

HNU MODEL PI-101 PHOTOIONIZATION DETECTOR

HNU MODEL PI-101 PHOTOIONIZATION DETECTOR

1. Turn the function switch to the battery check position. The needle on the meter should read within or above the green battery arc on the scaleplate. If the needle is in the lower portion of the battery arc, the instrument should be recharged prior to making any measurements. If red LED comes on, the battery should be recharged.

2. Turn the function switch to one of the three ranges. In this position the UV light source should be on. Look into the end of the probe to see the purple glow of the lamp.

3. Place the function/range switch in the "0-20" ppm position before entering the site.

C-4

MIE MODEL PDM-3 MINI-RAM AEROSOL MONITOR

MIE MODEL PDM-3 MINIRAM AEROSOL MONITOR

Operation

1. If the MINIRAM shows a blanked display, press OFF and wait until the display reads "OFF" before pressing MEAS to initiate measurement cycle or if the MINIRAM shows "OFF", press MEAS directly to initiate measurement cycle.

2. The functions performed by pressing each MINIRAM touch switch are as follows:

   A. MEAS

   The first readout displayed is "GO", followed by the last concentration reading or ".00". Approximately 36 seconds after pressing MEAS the fist new 10-second averaged concentration reading is displayed. All subsequent readings are concentration values in milligrams per cubic meter, updated every 10 seconds.

   The MINIRAM will now run in the measurement mode for 500 minutes (8 hours and 20 minutes), after which it will stop, displaying the OFF reading, retaining in storage the concentration average and elapsed time information.

   B. MEAS and TIME

   If both MEAS and TIME are pressed at the same time (press TIME first and while depressing it actuate MEAS) the MINIRAM will display "CGO" (for Continuous "GO"), and will then operate as above (i.e., pressing MEAS only), except that after the fist 8.3 hour run it will restart automatically and continue to measure for an indefinite number of 8.3 hour runs, (with the battery charger) until the OFF key is pressed, or until the batteries are exhausted at which time the MINIRAM shuts off automatically, displaying the "OFF" reading. Concentration averages and timing information for the last seven 8.3 hour runs will remain in storage at any given time.

   C. OFF

   When this key is pressed the MINIRAM will discontinue whatever mode is underway displaying "GCA" ("GCA" is displayed and printed out by the MINIRAM although the instrument is manufactured exclusively by MIE, Inc.) followed by the display segments check ("8.8.8=") and finally "OFF".

   If OFF is pressed during a measurement run the display will read "OFF" for 48 hours (unless another key is pressed during that period), after which the display will be blanked.

Every time the OFF key is pressed, during a measurement cycle, the MINIRAM will store the concentration average and elapsed monitoring time up to the time of that OFF command. The duration of the off period (up to 48 hours), i.e., between two consecutive measurement cycles, is also stored for each of up to 7 cycles.

OFF must be keyed before any other operation mode can be entered: setting ID#, zero referencing, playing back stored data, or changing the program code. Display functions, however, can be activated during the measurement mode.

D. TIME

During the measurement mode, if TIME is pressed the display will show the elapsed time, in minutes, to three significant figures, from the start of the last measurement run. The MINIRAM will automatically return to concentration display after the TIME key is released.

E. TWA

This key stands for Time-Weighted-Average. During the measurement mode, if TWA is pressed the display will indicate the average concentration in milligrams/m^3 up to that instant, from the start of the last run.

F. SA

This key stands for Shift-Average. During the measurement mode, pressing SA will provide a display of the aerosol concentration, up to that moment, averaged over an 8-hour shift period.

The value of SA is updated every 10 seconds. When releasing the SA key the MINIRAM display returns to the 10-second concentration display.

G. PBK

With the MINIRAM in the off mode (i.e., not in the measurement mode), the stored information can be played back by pressing PBK. If the PBK key is initially pressed the display will indicate "P" for one second. If PBK continues to be pressed for more than 1 second, then the stored data is automatically played back through the MINIRAM display: First, the identification number is displayed with the ID indicator bar on; next the shift or run number starting with the last run is shown; followed by the sampling time in minutes, for that run; followed by the off-time between the last and next run (in tens of minutes); finally, the average in mg/m^3. This sequence is repeated seven times. An average reading of 9.99 indicates that a significant overload condition occurred during that run. The total time required for the complete automatic playback on the MINIRAM display is approximately 70 seconds.

If PBK is pressed for less than one second "PA" will be displayed, and the stored data will be fed out through the digital output jack of the MINIRAM for printout, magnetic storage, telemetry, etc. a printout consists of 8 lines of data. The first 7 lines show the data for the last 7 measurement periods, and the last line shows the identification number (I), the programmable selection code (F), and the zero value for that data block (Z). In addition a check sum is printed out on a 9th line for modem/computer data transfer purposes. The first 7 data lines are subdivided into 4 columns. The first column identifies the measurement period (starting with the last or 7th); the next column lists the corresponding duration of each measurement period in minutes; the third column lists the off time between consecutive measurement periods, in minutes divided by 10; and the last column lists the average concentration values for each period in mg/m^3.

Either time-weighted, or shift average values can be printed, depending on the selected programmable code.

The speed of the digital transfer to a printer or other digital device can be user selected through the programmable selection code. for a 300 baud rate the transfer time for the stored data block is approximately 45 seconds.

H. ZERO

Pressing ZERO during a measurement period provides momentary display of the stored zero concentration value used by the MINIRAM to correct all digital concentration readings (the analog output signal is not zero-corrected). To update the ZERO value the MINIRAM must be in its off condition (press OFF in case of doubt). Then, press ZERO and wait until the display again indicates "OFF".

The average of 4 consecutive 10-second zero level measurements will then be stored by the MINIRAM as the new ZERO reference value. When operating the MINIRAM in high particle concentration environments (>5 mg/m^3) the zero value update should be performed approximately every 8 hours. At aerosol concentrations below approximately 1 mg/m^3 this update may only be required once a week, or even less frequently. The zero update should be performed in a clean-air environment.

I. ID#

Pressing ID# during a measurement period provides momentary display of the identification number stored with the MINIRAM memory.

The ID# key, in combination with other keys, is used for several additional programming functions described in the next section.

3. Programmable Functions

A. ID# Selection

In order to change the instrument identification number the MINIRAM must first be in the off mode (i.e., press OFF). Then press the ID# key, and the presently stored number (between 1 and 999) will be displayed, as well as the ID indicator bar. To increment the identification number press the ↑ key (same key as TWA), and to decrement the number press the ↓ key (same key at SA). Any number between 1 and 999 can thus be selected and will remain in storage until the batteries are disconnected, or if the MINIRAM is not recharged over a 6-month period.

Pressing the OFF key after the above identification number selection will remove the MINIRAM from the ID# selection routine and lock-in that number until a new number is selected. A complete ID# lock-out (i.e., a routine to preclude panel-control change of that number) can be accomplished by a separate programmable code selection.

B. Programmable Selection Code

The programmable code allows the user to panel-select several alternate functions and operating modes.

The program codes to select specific alternate operating modes area:

1	selects the alarm instead of ASCII digital output
2	selects the ID# lock-out
4	selects the TWA instead of the SA to be stored for playback
8	selects a 1-second pause after each printer carriage return (for slow printers)
32	selects 110 baud digital output rate instead of 300 baud
64	selects 600 baud digital output rate instead of 300 baud

These numbers are entered as a sum, e.g., to implement ID# lock out, TWA storage, and 1-second carriage return delay, the code number would be 14(2+4+8).

To enter the desired code (e.g., 14) follow these steps:

- Press OFF key and wait until "OFF" is displayed.
- Press ID# key and set program code to desired number (e.g., 14) by means of the ↑ and ↓ keys.
- Press TIME key (this will show previously entered code).
- Press ID# key again to lock in the new program code which will then be displayed.
- The preceding steps will cause the ID# to become equal to the programmable selection code. To restore the desired ID# (without affecting the selected code number which is now locked in, use the ↑ and ↓ keys again to select the ID# for the instrument.
- Press OFF to exit the ID# selection routine.
- To look at the programmed code number, at any time, start from the off condition, press ID#, then press TIME ("F" will then be displayed momentarily), after which the code number will be displayed. Press OFF to exit the code number routine.

If no specific alternate code is entered the MINIRAM will operate in its standard mode (equivalent to code 12) consisting of the following:

- ASCII digital output
- Panel-selectable ID number (preset to 999)
- Time-Weighted Average (TWA) values in memory storage
- 7-bit ASCII resolution
- 300 baud digital output
- Printer carriage return followed by a 1 second delay

B. If the ID# lock-out code has been selected then both the ID# and the programmable code can only be displayed (and printed out), but neither of the two can then be changed by means of the panel keys. In this case, in order to change the ID# if the lock-out code has been selected, or to alter the programmable code, the battery must be unplugged momentarily. Disconnecting the battery, however, causes the MINIRAM to lose all stored data, and cancels

all alternate program codes which may then be restored following the programmable selection code.

C. Alarm Level Adjustment

If the selected program code includes a 1, the MINIRAM will not provide an ASCII digital output but instead a switched output (at the digital output connector) which will close every time the measured 10-second concentration value exceeds a presettable threshold concentration level. If a 1 has been included in the code, then the ID# divided by 10 becomes the alarm level in milligrams/m^3. This level can be adjusted following the ID # selection procedure. For example, if an alarm level of 12.5 mg/m^3 is desired (and starting from the off mode), press ID#, adjust displayed number to 225 with the ↑ and ↓ keys, and press OFF. This number (e.g., 125) then becomes the ID# as well. It is not possible to enter a separate alarm level and ID# number.

gai

APPENDIX D

CALIBRATION PROCEDURES FOR AIR SAMPLING EQUIPMENT

D-1

TEI MODEL 43A PULSED FLUORESCENCE SO_2 ANALYZER

TEI MODEL 43A PULSED FLUORESCENCE SO_2 ANALYZER

Calibration:

1. Attach the output from the calibration gas to the bulkhead labelled "SAMPLE" on the rear bulkhead of the instrument. NOTE: The calibration gas should be delivered to the instrument at *atmospheric pressure*. It may be necessary to employ an atmospheric dump bypass plumbing arrangement to accomplish this.

2. Slide the electronics section of the instrument forward. Locate the third printed circuit card from the front of the instrument (Timing PC Board). This PC card has a three-position toggle switch (M-L-H) and a high voltage adjust potentiometer. The three-position switch is the coarse gain for the photomultiplier (PMT) high voltage, while the potentiometer is a fine gain adjustment. This control will be used for setting the instrument gain for calibration purposes.

3. Connect zero air source to bulkhead on rear of instrument labelled "ZERO". Switch the instrument to the "ZERO" mode. Switch front panel "ZERO" control potentiometer until instrument reads zero. A five percent recorder offset is recommended to facilitate this adjustment.

4. Turn input mode switch on front panel of instrument to "SAMPLE", allowing calibration gas to flow into instrument.

5. Allow instrument to come to a stable reading (about five minutes).

6. Adjust PMT high voltage using three-position toggle switch and potentiometer on Timing PC Board. Adjust PMT high voltage so that instrument indicates calibration gas concentration plus or minus 0.010 ppm.

7. Turn input mode switch on front panel of instrument to "ZERO".

8. Allow instrument to come to a stable reading.

9. Since Items 3 through 8 are iterative, return to Item 3 and proceed until no adjustment in the PMT high voltage is required to have instrument read calibration gas concentration plus or minus 0.010 ppm in Step 8.

10. Adjust front panel "ZERO" control potentiometer until instrument reads "ZERO"; lock in place.

11. Turn mode switch on front panel of instrument to "SAMPLE".

12. Allow instrument to come to a stable reading (approximately 10 minutes).

13. Adjust "SPAN" control potentiometer on front panel electronics section so that instrument reads exactly the calibration gas concentration.

14. Lock "SPAN" control potentiometer at setting.

15. Connect SO_2 calibration gas with concentration below initial calibration gas to bulkhead on rear of instrument labelled "SAMPLE".

16. Record instrument reading after allowing time for instrument to stabilize.

17. Repeat Items 15 and 16 for five different concentration points.

18. Turn front panel mode switch to "ZERO". Allow instrument to come to a stable reading and record as zero.

19. Plot a graph of instrument reading against SO_2 concentration. This is the instrument calibration curve.

20. Remove calibration gas from bulkhead on rear of instrument labelled "SAMPLE".

21. Attach sample gas to be measured to bulkhead on rear of instrument labelled "SAMPLE".

22. Turn front panel mode switch to "SAMPLE".

23. Instrument is now ready for sample gas measurements.

D-2

TEI MODEL 45 PULSED FLUORESCENCE H_2S ANALYZER

TEI MODEL 45 PULSED FLUORESCENCE H_2S ANALYZER

Calibration:

1. Calibrate the TEI Model 43A Pulsed Fluorescence H_2S Analyzer as described in Section D-1.

2. Set the switch on the Model 45 to the SO_x setting. Turn input mode switch on front panel of Model 43A to "SAMPLE".

3. Attach first calibration gas to bulkhead on rear of instrument labelled "SAMPLE". Record instrument reading after allowing time for instrument to stabilize.

4. Repeat Item 3 for three different concentration points.

5. Plot a graph of instrument readings against H_2S concentration. This is the instrument calibration curve.

6. Remove calibration gas from bulkhead on rear of instrument labelled "SAMPLE".

7. Attach sample gas to be measured to bulkhead on rear of instrument labelled "SAMPLE".

8. Instrument is now ready for sample gas measurement.

D-3

NATIONAL DRAEGER MODEL 190 DATA-LOGGER GAS MONITOR

NATIONAL DRAEGER MODEL 190 DATA-LOGGER GAS MONITOR

Calibration

Zero Adjustment

1. Use red function key to place unit into the constant concentration mode (Refer to Figure 2, Appendix C-4). The instrument must be zeroed utilizing the calibration adaptor with a nitrogen-cylinder (P/N 4594838) or in a clean air environment. (Refer to the Span Adjustment Section for cylinder operation only).

2. Locate the Zero and Span adjustments. Refer to Figure 1. Place the calibration adaptor over the filter housing and allow the nitrogen to flow for approximately two (2) minutes.

3. Adjust the zero potentiometer until the display reads zero ppm. Making sure the zero potentiometer is capable of adjusting the reading ± 5 ppm. If not, refer to the Troubleshooting Section. Remove the calibration adaptor and nitrogen (if used).

Span Adjustment

4. Prepare the span gas cylinder and attach the calibration adaptor assembly to the cylinder regulator. Turn on the regulator (flow rate: 200 cc/min) and purge the calibration adaptor assembly for approximately one (1) minute).

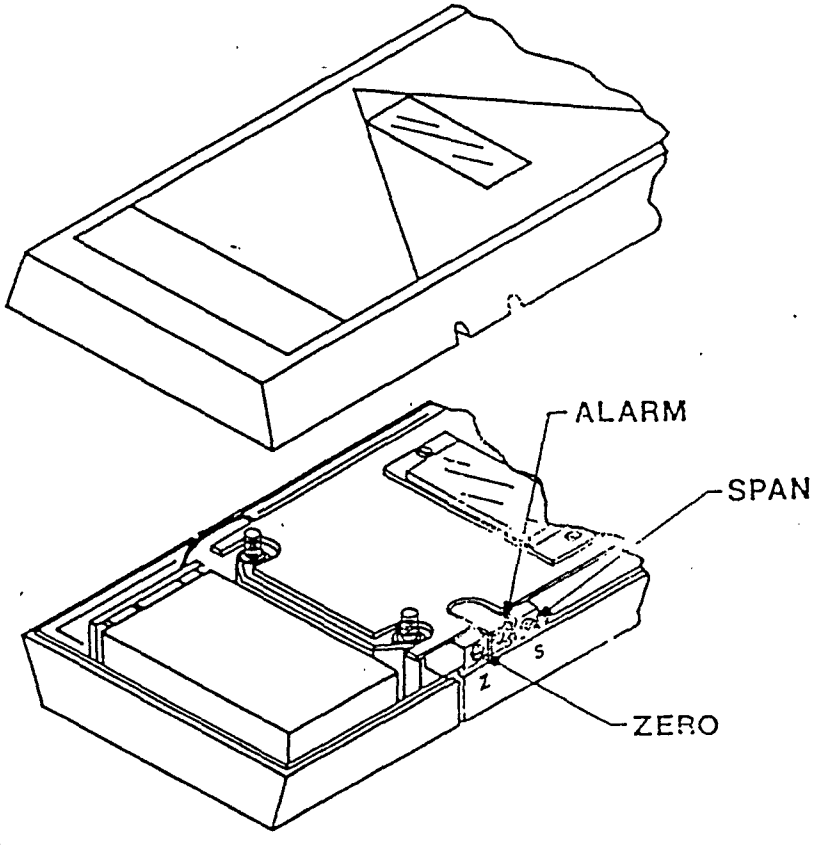
5. Place the calibration adaptor over the filter housing (Refer to Figure 2).

6. Wait approximately two minutes or until the display is showing a constant reading. New or unused sensors may take longer to stabilize. Calibration should not be performed in areas of high air velocity or turbulence. High air flow around the calibration adaptor and can penetrate through the holes in the sides of the calibration adaptor and cause unstable readings Insure that air flow (from fume hood ventilation systems, etc.) does not cause excessive turbulence around the calibration adaptor. If unstable readings occur, refer to the Troubleshooting Section.

7. Adjust the span potentiometer until the display indicates the ppm value of the known calibration gas.

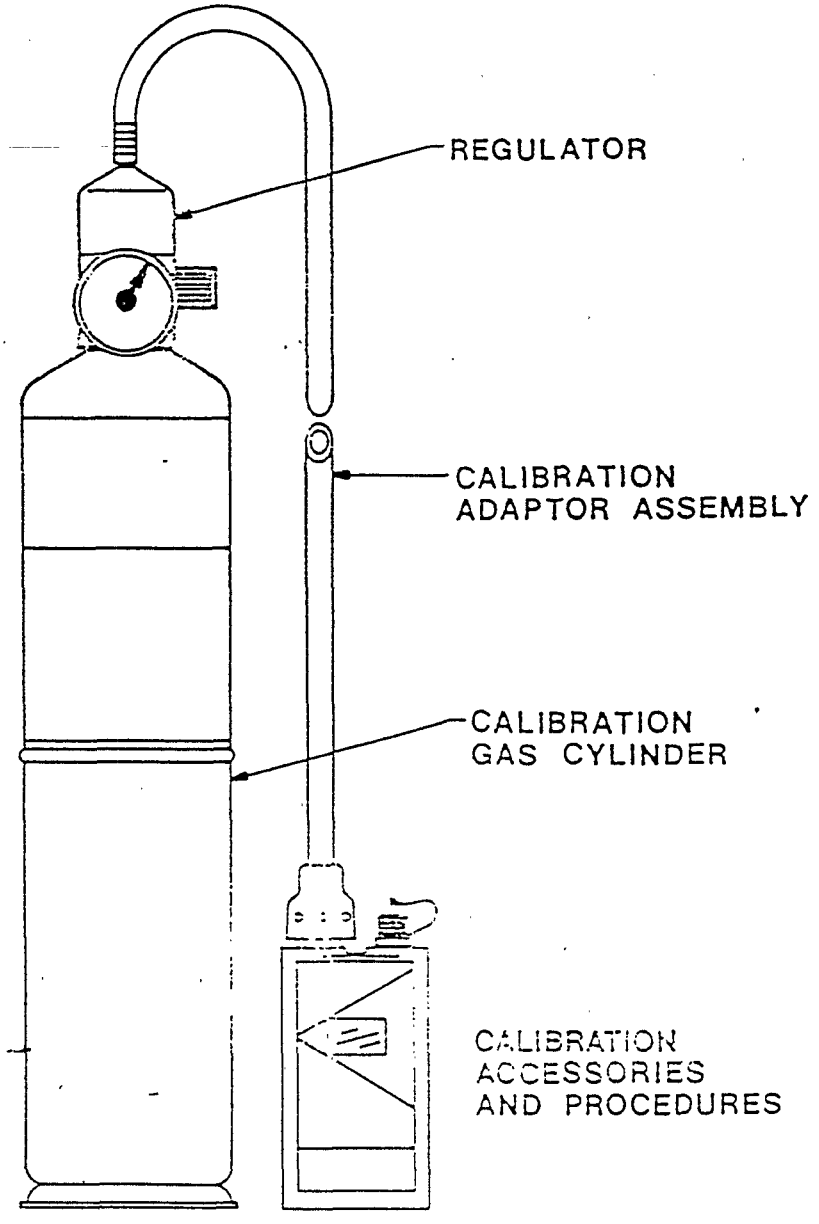
8. Perform alarm set-point adjustment, if necessary.

Figure 1



ALARM ADJUSTMENTS

Figure 2


REGULATOR
CALIBRATION
ADAPTOR ASSEMBLY
CALIBRATION
GAS CYLINDER
CALIBRATION
ACCESSORIES
AND PROCEDURES

Alarm Adjustment

1. Remove the filter housing from the unit.

2. Loosen the three (3) screws and remove the top housing.

3. Locate the zero and alarm potentiometer. Refer to Figure 1.

4. Adjust the zero potentiometer until the display indicates the ppm value of the desired alarm-set point.

5. If alarm is sounding, adjust alarm set potentiometer clockwise until alarm is quieted and then counterclockwise until alarm sounds.

   If alarm is quiet, adjust alarm set potentiometer counterclockwise until alarm sounds.

6. RESET ZERO POTENTIOMETER TO 0.

7. Replace the top housing. Be sure that the battery leads are routed around the screw post. Tighten the three (3) screws.

8. Replace the filter housing. The monitor is ready for use.

gai

D-4

HNU MODEL PI-101 PHOTOIONIZATION DETECTOR

HNU MODEL PI-101 PHOTOIONIZATION DETECTOR

Calibration

1. Before attaching the probe, check the function switch on the control panel to make sure it is in the off position. The 12 pin interface connector for the probe is located just below the span adjustment on the face of the instrument (see Figure 1). Carefully match the Alignment Key in the probe connector to the 12 pin connector on the control panel, and then twist the probe connector until a distinct snap and lock is felt.

2. Insert the HNu probe into one end of a T connection. Insert the second end of the T connection into the calibration gas cylinder fitting. Use the mid-range (20 to 200 ppm) calibration gas first. The third end of the connection should be left open.

3. Set the function switch to the 20 to 200 ppm range.

4. Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotameter. The instrument will draw in the volume required for detection, with the open end of the T connection releasing the excess flow.

5. Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas (calibration gas valve is labeled on the cylinder).

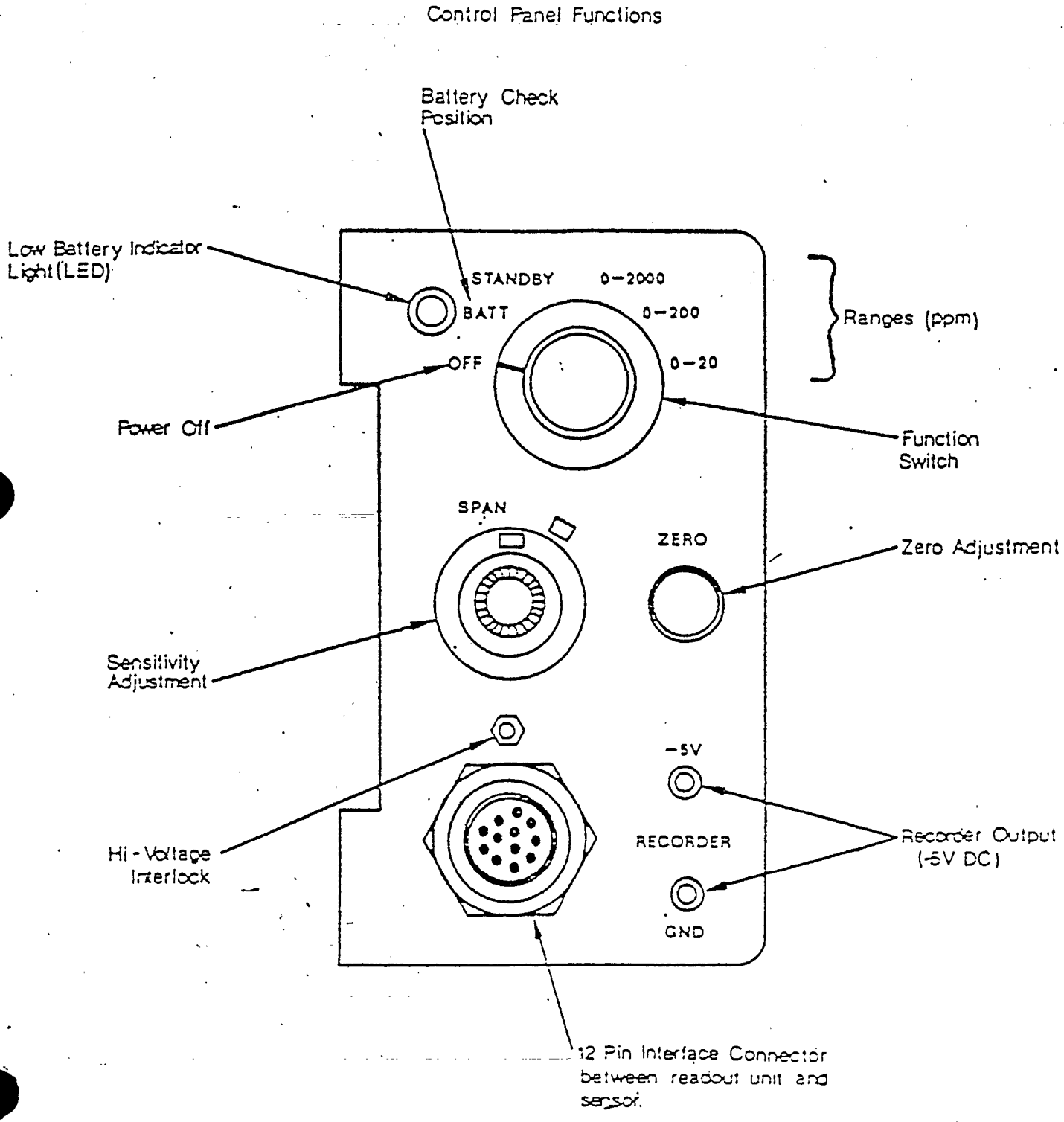
6. Turn instrument switch to the standby position and check the electron zero. Reset zero potentiometer as necessary.

7. Record all original and readjusted settings.

8. Next, set the function switch to the 0 to 20 ppm range. Remove the mid-range (20 to 200 ppm) calibration gas cylinder and attach the low-range (0 to 20 ppm) calibration gas cylinder as described above.

9. Do not adjust the span potentiometer. The observed reading should be ± 3 ppm of the concentration specified for the low-range calibration gas. If this is not the case, recalibrate the mid-range scale repeating steps 1 to 7 listed above. If the low-range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. When the observed reading is within the required tolerances, the instrument is fully calibrated.

Figure 1



AR303909

D-5

MIE MODEL PDM-3 MINI-RAM AEROSOL MONITOR

MIE MODEL PDM-3 MINI-RAM AEROSOL MONITOR

Calibration

1. Place MINIRAM in a clean environment (e.g., air-conditioned office).

2. Remove battery pack.

3. Disconnect battery connector (remember that all stored data will thus be lost/erased from MINIRAM memory).

4. While leaving battery pack lying next to MINIRAM, re-connect the two units (i.e., plug in connector).

5. Immediately observe MINIRAM display. It will be performing a slow segment-by-segment display checkout. As soon as it displays ".00", press OFF, thus interrupting the initial automatic zero check. Wait until the display indicates "OFF" and then press MEAS and wait approximately 36 seconds.

6. Observe 10-second readings (typically the range of 1 to 3 mg/m^3) and record manually a few consecutive readings. Calculate the average of these values.

7. Identify small potentiometer screw (visible through an opening in the foil shield of the open MINIRAM) opposite the digital output jack. Adjust this potentiometer, using a fine screwdriver, until the average MINIRAM reading is increased or decreased (with respect to the average obtained in 8.6) by the desired ratio (e.g., as determined by previous gravimetric comparison runs).

8. Shut off MINIRAM, reposition and secure battery pack, and re-zero instrument as usual. All subsequent concentration readings are now corrected by the desired ratio.

APPENDIX E

LANCASTER LABORATORIES, INC. QUALITY ASSURANCE PLAN

SECTION 4
QUALITY ASSURANCE PROGRAM

The major function of an independent laboratory in today's society is to generate technical information. In our case, the information consists of the results of chemical and microbiological analyses, along with whatever auxiliary information is necessary for proper interpretation, and research reports.

Our clients use this information for a variety of purposes. It may be used to demonstrate compliance with a government regulation; to evaluate a raw material for a manufacturer or food processor; to demonstrate value or quality of a finished product, as in nutritional analyses; to establish the basis for a patent; or to decide a legal dispute. This information has a high and intrinsic value over and above the cost of providing it to a client. Since this information is important to our clients, it is necessary to produce it under a program which will assure that it has the necessary "quality," i.e., that it has a degree of accuracy commensurate with its intended use. This section of the manual will describe the Quality Assurance Program under which we operate. Details relating to specific types of technical operations will be found in the sections dealing with the various technical groups.

A. OBJECTIVES OF THE QUALITY ASSURANCE PROGRAM

The objectives of our Quality Assurance Program, as directed by Corporate Management are:

- To establish quality control procedures which will ensure that data generated in the laboratory are within acceptable limits of precision and accuracy.
- To establish procedures to document that these quality control measures are, in fact, being carried out.
- To establish procedures to ensure the "accountability" of the data, i.e., that the results reported do apply to the sample as submitted.
- To establish procedures to ensure that any result reported to a client is traceable to:
    - The date the analysis was run
    - The analyst who performed the test
    - The raw data generated during the performance of the test
    - The condition of any instrument or equipment at the time it was used in the test
    - The status of the quality control system at the time of the test
- To establish procedures which minimize the possibility of loss, damage, or tampering with the data.

The administration of our Quality Assurance Program is the responsibility of the Quality Assurance Director in cooperation with the Division Directors and Group Leaders. The Quality Assurance Director reports directly to the President, thus ensuring that corrective actions can be taken immediately for any quality problems. The Quality Assurance Director does not have any direct supervisory responsibility for the generation of technical data to avoid any "conflict of interest" which could interfere with the Quality Assurance Program.

The formal structure of our Quality Assurance Program is described in a set of Standard Operating Procedures (SOPs). Copies of these SOPs are given to each Group Leader and they are available to all laboratory personnel. The following is a list of our current Quality Assurance (QA) SOPs, with a brief description of each:

QA-101 Sample Collection - In order for meaningful analytical data to be produced, the samples must be representative of the system from which they are drawn. Our samplers are trained and use written sampling procedures. Sample containers are chosen according to the analyses to be performed and are labelled to fully identify each sample. Where necessary, chemical preservatives and temperature adjusted storage during transport to the laboratory are employed.

QA-102 Sample Log-in - To ensure accountability of results, each sample is assigned a unique sample identification number as soon as possible after its receipt at the laboratory. Information corresponding to sample identity is logged with this sample number.

QA-103 Sample Storage and Disposal - Because sample integrity can be compromised by improper storage, samples are maintained in various locations to prevent deterioration. Locations are assigned in refrigerators, freezers, and at room temperature to assure that the chemical, physical, and biological properties of samples do not change prior to analysis. Sample locations are tracked by computer to prevent loss. After results are reported to clients, samples may be held for a period of time in case additional testing is required. Samples are then disposed of or returned to the client.

QA-104 Chain-of-Custody Documentation - Samples being tested for litigation or regulatory purposes may require locked storage and documentation of the laboratory personnel who used the sample as well as the times during which the sample was removed from locked storage. Chain-of-custody documentation minimizes the possibility of tampering with the samples, and is available upon request.

QA-105 Analytical Methods Manual - The method manuals, which are comprised of clear, complete written instructions for performing each standard test, are the basis for our analytical testing program. In addition to the actual procedure, each method includes safety and quality control information. Every standard method is assigned a unique identification number to ensure traceability. Copies of the methods are readily available to analysts in the lab.

QA-106 Validation and Authorization of Analytical Methods - Although our routine testing procedures are based on official or standard methods whenever possible, laboratory personnel must verify that acceptable precision and accuracy are obtainable before management will approve the use of any method. Validation studies may include the use of standard reference materials, fortified samples, or replicate analyses.

QA-107 Analytical Methods for Non-standard Analyses - Frequently, clients request analyses which are not regularly performed in our laboratory although we have the technical capabilities to do so. In these instances, methods may be supplied by the client, dictated by regulations, derived from scientific literature, or developed in the laboratory. In any case, special procedures must be followed to ensure that the method used is thoroughly documented. In addition, records of validation or quality control samples performed are maintained.

QA-108 Subcontracting - For the convenience of our clients, some analyses which we do not perform may be subcontracted to other laboratories. These laboratories must submit evidence of their qualifications to us. Wherever possible, laboratories certified by appropriate agencies will be used. Results produced by subcontracted laboratories will always be designated as such on our laboratory reports.

QA-109 Laboratory Notebooks - All data which is manually acquired in the laboratory is entered into bound notebooks. Distribution of notebooks to personnel is controlled, with a master file kept of the recipient of the notebook, date issued, date returned, and place of storage of completed notebooks. Personnel are instructed in the proper method of data entry. All entries are made in ink, dated, and signed. Blank pages or substantial portions of pages are cancelled out. Periodic review and signing of notebooks by supervisors is also required.

QA-110 Reagents - Chemical and reagent quality will have a significant impact on our analytical results. Procedures for receiving, preparing, documenting, and storing reagents have been set forth to ensure that only supplies of adequate quality are used in our testing. Each analytical method contains specifications for the required materials.

QA-111 Instrument and Equipment Calibration - All instruments and other equipment are calibrated on a regular basis in accordance with written procedures, with frequency of calibration dependent on the type of instrument and its frequency of use. Acceptable limits of accuracy are also described in the procedures.

QA-112 Instrument and Equipment Maintenance - All maintenance work done on instruments and equipment is recorded in bound notebooks, with separate notebooks kept for each instrument. Routine, preventive maintenance is performed according to written procedures.

QA-113 Data Entry and Verification - In order to minimize errors, data reduction and transcription is computerized wherever possible. Analysts responsible for the generation of data also have the responsibility to log their raw results into the computer for reporting to clients. Results are then reviewed by a supervisor or the supervisor's designee before reporting to clients.

QA-114 Data Storage and Security - The data generated in our laboratories is a valuable commodity, purchased by our clients. In order to provide traceability, data is stored in an orderly fashion under controlled access so that loss, deterioration, or tampering is prevented. Copies of reports and all supporting raw data will be maintained for ten years.

QA-115 Quality Control Records - The term "quality control" is used to denote those laboratory procedures whose purpose is to ensure that the analytical system is in control within established limits of accuracy and precision. Quality control procedures for each analysis are documented in the written method. Results of quality control analyses are subjected to statistical evaluation to detect trends and outliers. Documentation of quality control samples includes a monthly report of the actual results obtained and the established acceptance limits.

QA-116 Investigation and Corrective Action - One of the most effective means for maintaining the production of high quality data is to respond immediately to indications of suspicious data or equipment malfunctions. Whenever results from quality control samples fall outside of established limits, the cause of this irregularity is investigated and corrected as soon as possible. Documentation of these activities is then used to prevent future occurrences of the problem.

QA-117 Personnel Training Records - Supervisors are required to maintain personnel training records of all personnel under their supervision. Training records for technicians indicate the date on which they were considered to be proficient in various laboratory techniques or in various analytical methods. Training records for professionals indicate special training or education received over and above their educational qualifications.

QA-118 Quality Assurance Audits - To ensure that laboratory personnel are adhering to the procedures set forth in our Quality Assurance Operations Manual, periodic checks of each group are made by the Quality Assurance Department. These audits may entail observation as procedures are carried out or a review of records to demonstrate traceability and compliance with all documented recordkeeping procedures. Reports of the findings of these audits are made to management.

QA-119 Proficiency Samples - The choice and frequency of quality control samples are specified in the analytical method and are often based upon the recommendations of regulatory agencies such as EPA. In addition, samples are obtained from various organizations that conduct collaborative studies or provide reference materials. Quality control samples are also submitted blind to analysts so that they may be analyzed without any bias which may be introduced by known quality control samples.

QA-120 Documentation of Programming for the Sample Management System - The sample management system is used to perform calculations which convert raw data to the final result reported to our clients. Thus the computer code is part of the chain of documentation relating to each sample and must be recorded. All new or modified programming on this system is tested prior to use in the laboratory.

QA-121 Computer Validation - The design and development of software systems used to acquire and process data in the laboratories is a responsibility that is distributed among various groups within our laboratory. To maintain consistency of documentation and to ensure that all programs function correctly, guidelines for development, validation, and maintenance of quality computer software are provided. These guidelines are consistent with GLP requirements.

B. ACCOUNTABILITY OF RESULTS

The term "accountability" as applied to testing results refers to the procedures taken to assure that the data reported refers to the sample submitted. In other words, that there has been no sample mix-up and the sample has been properly handled in its transit through the laboratory to minimize changes in chemical composition or bacteriological quality.

At Lancaster Laboratories, Inc., we rely on our computerized Sample Management System to track and control movement of samples from the time of receipt until disposal. The system works as follows:

Samples are received at the laboratory in one of three ways: by personal delivery, by mail or common carrier, or by sample pick-up by laboratory personnel. All samples are received by personnel of the Sample Administration Group, who are responsible for sample log-in and tracking. The first step, after sample receipt, is its placement in our Sample Management Program. All samples are logged into the computer along with pertinent information, e.g., the client's name, account number, client designation or description, and analyses requested. The computer assigns a unique number to the sample and requests information regarding necessary storage conditions, e.g., refrigeration, freezing, etc. The computer then assigns a storage location number which designates where the sample will be kept until analysis is begun and prints a label which is immediately attached to the sample container. The information contained on the label includes sample number, client designation, tests scheduled, and a bar code. The computer also assigns a storage time or disposal date which varies according to the nature of the sample or with specific instructions from the client.

The data base, which is thus automatically generated, is used in many ways. For example, each Group Leader receives a daily printout, which lists all samples and analyses waiting to be run in the laboratory. This is of inestimable value in planning and organizing the workload. The date of sample collection is also available through the computer to ensure that holding times are met, where applicable.

When a sample is to be analyzed, it is retrieved from the designated storage space by the analyst or by a member of the Sample Support Group if storage is in a controlled-access area. During analysis, raw data is recorded in ink in bound notebooks or on print-outs from instruments and then entered into the computer against sample number and analytical method number. Some instruments are connected directly to the Sample Management System, eliminating manual transcription. The computer performs many of the calculations to avoid a common source of error. When all analyses are completed and have been verified by a supervisor or designate, the computer generates a report. The client receives a copy of the face of the report containing the results of the analysis plus comments entered by the Group Leader or the analyst where necessary. The back of the laboratory copy of the report contains the raw data plus the names of the analysts who made each entry. This copy is retained in our archives.

The following page is a copy of a simulated report. Note that opposite each reported result is a code number, which identifies the analytical method used. In addition, to avoid ambiguity in interpreting results, the reverse side of the client's copy of the report contains an explanation of all symbols and units used in reporting data. The report also contains the name of the Group Leader who reviewed the final report.

In the case of samples which are likely to be involved in litigation or a legal dispute, more stringent sample handling procedures are available upon request. Strict chain-of-custody procedures are followed. The delivery of the sample is documented, the sample is kept under locked storage, and photographs or videotapes may be used to document the condition of the sample or visual aspects of the test. After analysis, samples will be returned to the client or disposed of at his/her written request.

ANALYSIS REPORT

Lancaster Laboratories INCORPORATED

WLK1586D-14

LLI Sample No. WW 1335799

Smith Engineering, Inc.
1000 Any Street
Lancaster, PA 17601-5994
Water Sample from Monitoring Well #5
Collected on 12/8/90 at 1547 by MLH

Date Reported 12/16/90
Date Submitted 12/08/90
Discard Date 01/16/91
Collected by MLH
P.O.
Rel.

ANALYSIS CODE	RESULT AS RECEIVED		LIMIT OF QUANTITATION	LAB
Total Coliform	< 2.2	/100ml	2.2	030301500
Nitrite Nitrogen	0.05	mg/l	0.02	021900800
Nitrate Nitrogen	11.	mg/l	0.05	022000700
Ammonia Nitrogen	4.1	mg/l	0.1	022202600
Ortho-Phosphate as P	2.1	mg/l	0.05	022601100
Total Organic Carbon	8.5	mg/l	0.5	027302500

The Total Organic Carbon (TOC) result reported above was determined by measuring total carbon by a persulfate digestion/infrared detection method on an acidified sample which has been purged of inorganic carbon using nitrogen. It represents "nonpurgeable TOC."

Pesticides/PCB's		attached		017819500
Lead	0.25	mg/l	0.05	025501200
Trichloroethene	12.	ug/l	1.	041800500

1 COPY TO Smith Engineering, Inc. ATTN: John Smith

Questions? Contact Environmental Client Services at (717) 656-2301
00649 10.00 2700

Respectfully Submitted
Lancaster Laboratories, Inc.
Reviewed and Approved by:

Bethany Ebling
Group Leader, Water Quality

See Reverse Side For Explanation Of Symbols And Abbreviations And Our Standard Terms And Conditions

31

AR303918

C. QUALITY CONTROL

1. Analysis of Blank, Spiked, Duplicate, Reference, and Control Samples

Each specific analytical method includes the particular quality control requirements performed to ensure that the data produced is of known quality. In addition to the necessary calibration standards, these quality control checks may consist of one or several different types of checks. Blanks, spikes, duplicates, reference materials, and control samples are employed, as appropriate for the analysis. The general criteria for the use of each of these types is outlined below.

Blanks are analyzed with most types of analyses to prevent reporting of false positives caused by factors in the laboratory system. Blanks are prepared and analyzed using the same reagents and equipment as the samples in the batch the blank represents. Acceptance criteria for blanks is based on a multiple of the laboratory detection limit.

Spiked samples are samples fortified with a known amount of target analyte and subjected to the entire analytical procedure. The recovery of the method is calculated and indicates the appropriateness of the method for the matrix. Many of the gas chromatographic analyses in Environmental Sciences employ the special category of quality control samples known as matrix spike duplicates. This is the analysis of a pair of fortified samples from the same source. The use of matrix spike duplicates yields precision information as well as recovery. The acceptance criteria for percent recovery on spiked samples is based on EPA or other agency recommendation or past information generated in our laboratory.

Duplicate sample analysis is the analysis of the same sample twice in order to determine the precision of the analysis. The relative percent difference (RPD) between the two determinations is calculated and compared to values prescribed by EPA or determined by statistical analysis of past information generated in our laboratories.

Reference materials are samples which contain a known amount of target analyte. These are routinely analyzed to ensure accuracy of the analytical procedure. The reference materials may be from NBS or EPA, or they may be prepared in our own laboratories. Accuracy information determined from reference materials is valuable because variables specific to sample matrix are eliminated. The acceptance criteria for this type of quality control is either dictated by the agency from whom the material is obtained or by statistical analysis of past information generated in our laboratories.

Control samples are similar to reference materials except that the true value of the target analyte is not known with specific degrees of confidence. Acceptance criteria is developed by applying statistical techniques to repetitive determinations.

In addition, many of our chromatographic analyses employ surrogate and internal standards to evaluate analytical efficiency. The acceptance criteria for the recovery of these compounds is the same as that listed above for spiked samples.

The results of all quality control samples are entered into the computer in the same way as the results of client samples. The computer is programmed to compare the individual values with the acceptance limits and inform the analyst if the results of the quality control tests are in or out of specification. If the results are not within the acceptance criteria, corrective action suitable to the situation must be taken. This may include, but is not limited to, checking calculations, examining other quality control analyzed with the same batch of samples, qualifying results with a

comment stating the observed deviation, and reanalysis of the samples in the batch. Daily reports of quality control outliers are generated by the computerized system to keep management informed. The cause and solution to the problem is documented to prevent reoccurrence. In addition, computerized reports on the results for all quality control analyses including mean and standard deviation are generated monthly. These are used by the Quality Assurance Department to check for trends which may indicate method bias. Control charts are plotted via computer and may be accessed at any time by all analysts.

These programs have been found to be invaluable in monitoring our analytical procedures and detecting situations where the system was tending to lose control before it became serious enough to affect the integrity of the sample results.

2. **Blind Samples**

For many of the more common analyses performed in our laboratories, the Quality Assurance Department periodically submits blind samples to the laboratory using a pseudo-client name. These blind samples are similar to control samples, and results are reported directly to the Quality Assurance Department, for data evaluation. Summaries of the data are prepared periodically and reported to the Group Leaders and Division Directors. The value of the blind sample system lies in the fact that the analysts are unaware that they are analyzing a quality control sample, thereby avoiding unconscious bias in performing the analysis.

3. **Proficiency Sample Testing**

Proficiency samples and check samples are samples submitted to the laboratory by an outside organization. The concentration of certain analytes is known to the outside organization but not to the laboratory. The laboratory is required to analyze the samples for the indicated analytes.

The proficiency sample testing programs are part of an accreditation process, and results of samples must be within limits defined as acceptable by the submitting organization, or the laboratory may lose its accreditation. In the case of check sample programs, participation is voluntary, and results are used to compare the laboratory's competence to other participating laboratories. The following is a list of proficiency testing and check sample programs in which Lancaster Laboratories, Inc. participates:

Organization	Sample Type	Analytes
U.S. Environmental Protection Agency	Potable water	Metals, organics, THMs, nitrate and fluoride, VOCs
U.S. Environmental Protection Agency	Wastewater	Various pollutants
National Institute for Standards & Technology (NVLAP)	Bulk building materials	Asbestos
American Industrial Hygiene Association	Filters and charcoal tubes	Metals, organics, and asbestos fibers

Organization	Sample Type	Analytes
U.S. Department of Agriculture	Adipose tissue	Pesticides and PCBs
U.S. Department of Agriculture	Meat (split samples)	Moisture, protein, fat, and salt
PA Department of Agriculture	Dairy products	Bacterial contamination
American Association of Feed Control Operations	Animal feeds	Moisture, protein, fat, fiber, ash, phosphorus, calcium, and various additives (nutritional and veterinary drugs)
American Oil Chemists	Vegetable oils	Fatty acid profile
New York Department of Health	Non-potable water	Various organic and inorganic pollutants
American Association of Cereal Chemists	Cereal	Vitamins, minerals and proximate, and micro-biological contamination